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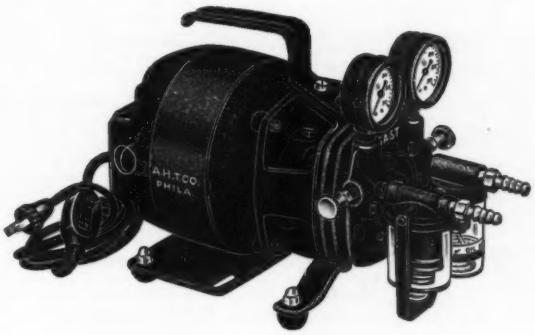

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This book is a systematic survey of the various branches of the surface chemistry of solids. It deals with all those chemical and physico-chemical phenomena which relate to the activity of the surface of a solid, or to the interface between a solid and a gas, a liquid or another solid. The author treats many of these phenomena, such as the adsorption of gases and vapours, the spreading of a liquid over a solid, catalysis and adhesion, with a consistent viewpoint as far as practicable. He summarizes and evaluates the theories of leading workers in these fields. Emphasis throughout is on general principles in order to simplify an exceedingly complex subject for the benefit of students and others who are not well versed in physical chemistry.

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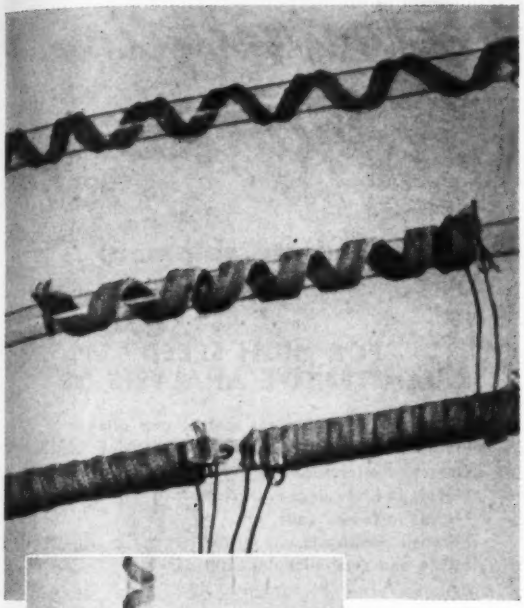

for laboratory applications and for use in pilot and production plant

These tapes consist of one or more fabric bands of resistance wire separated and bordered by bands of high temperature resisting glass fibre yarn. The width and length of the various tapes are carefully determined by the current carrying capacity of the resistance wire. The surface loading of standard heating tapes has been arranged approximately at 2½ watts/inch² (0.4 watts/cm²) area of tape. This wattage concentration enables temperatures of 400 °C. and over to be reached inside a glass tube of 2 mm. wall thickness.

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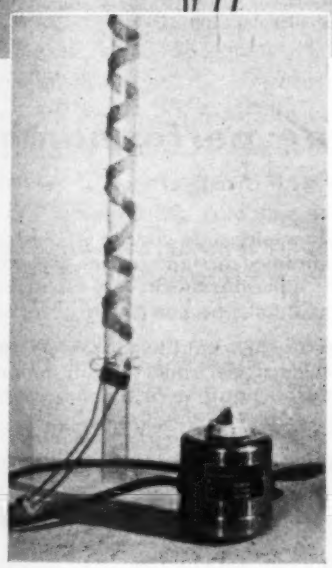

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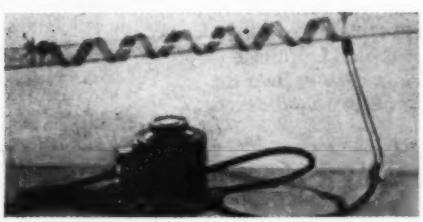

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HT.104	"	180	89	500	2-6	19.25
HT.201	Insulated one side	90	25	100	0-8	11.40
HT.202	"	125	64	250	1-5	14.90
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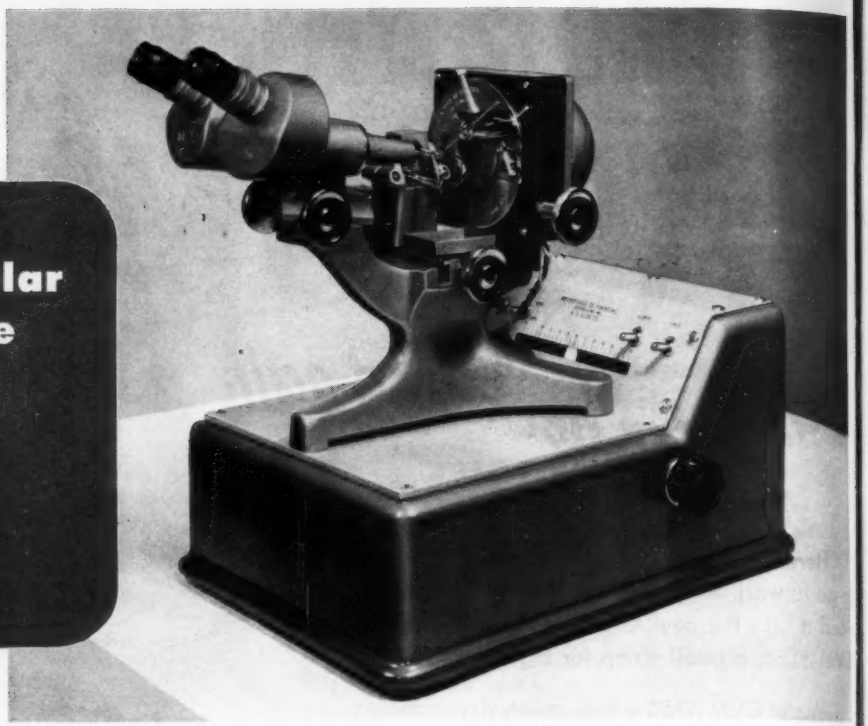

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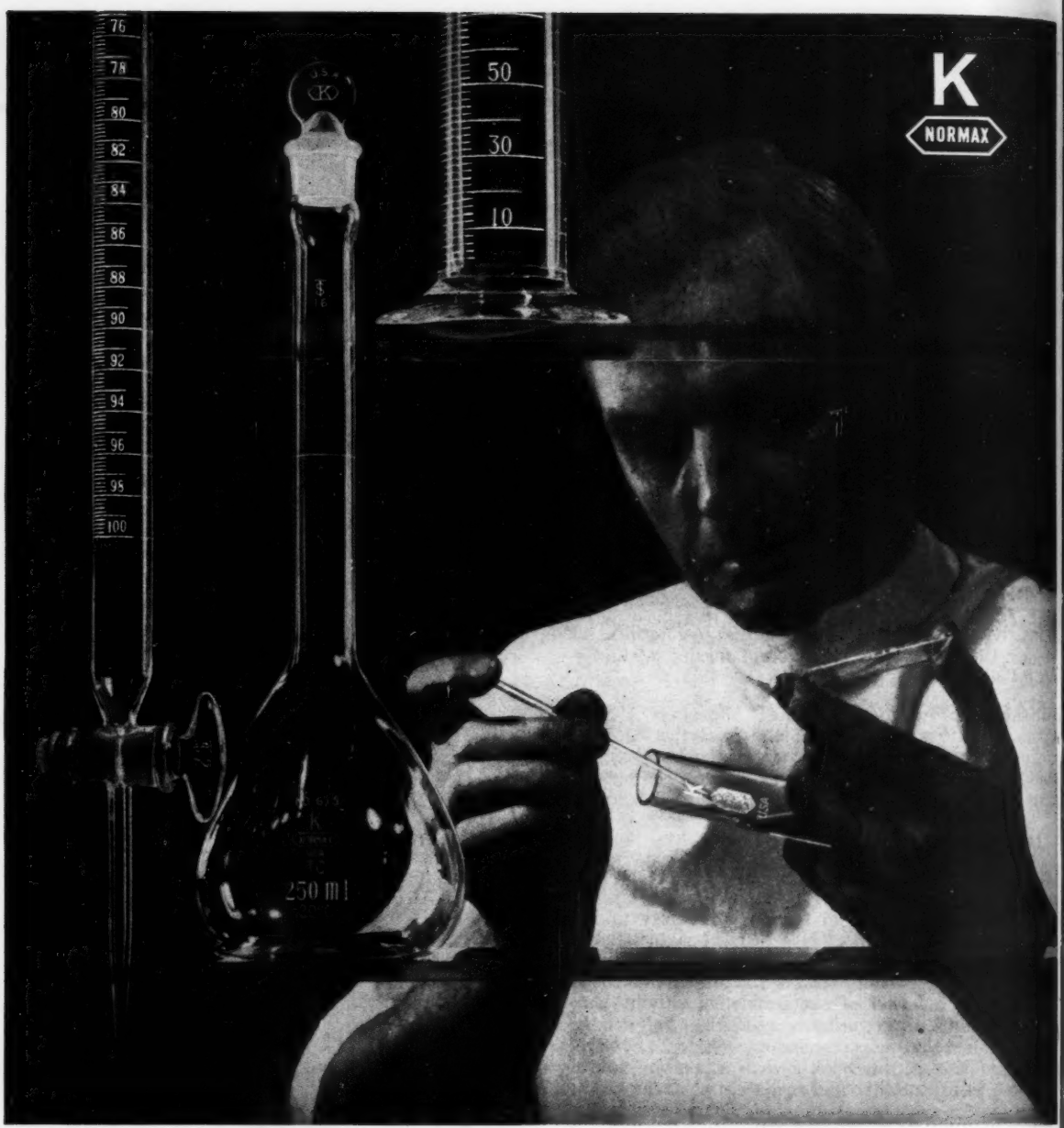

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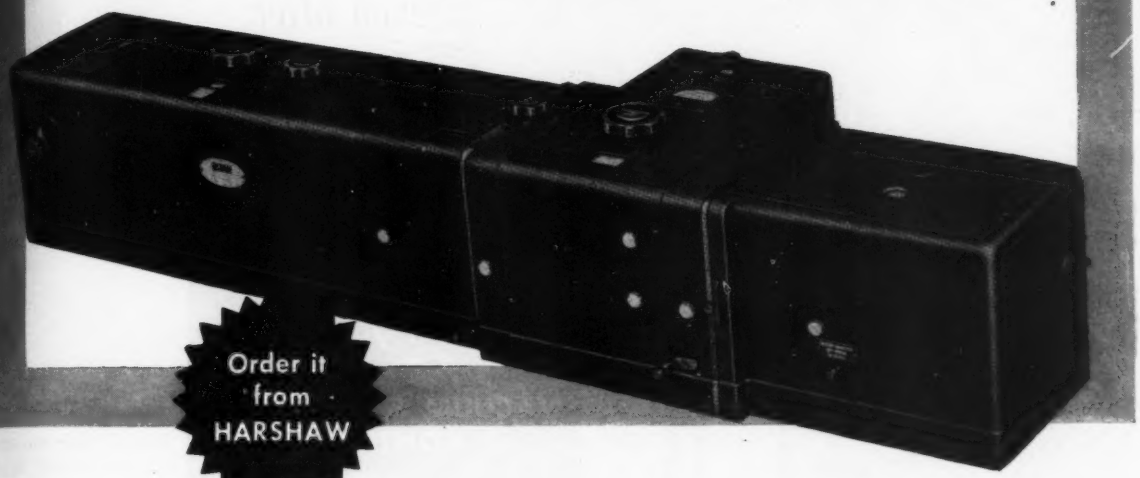

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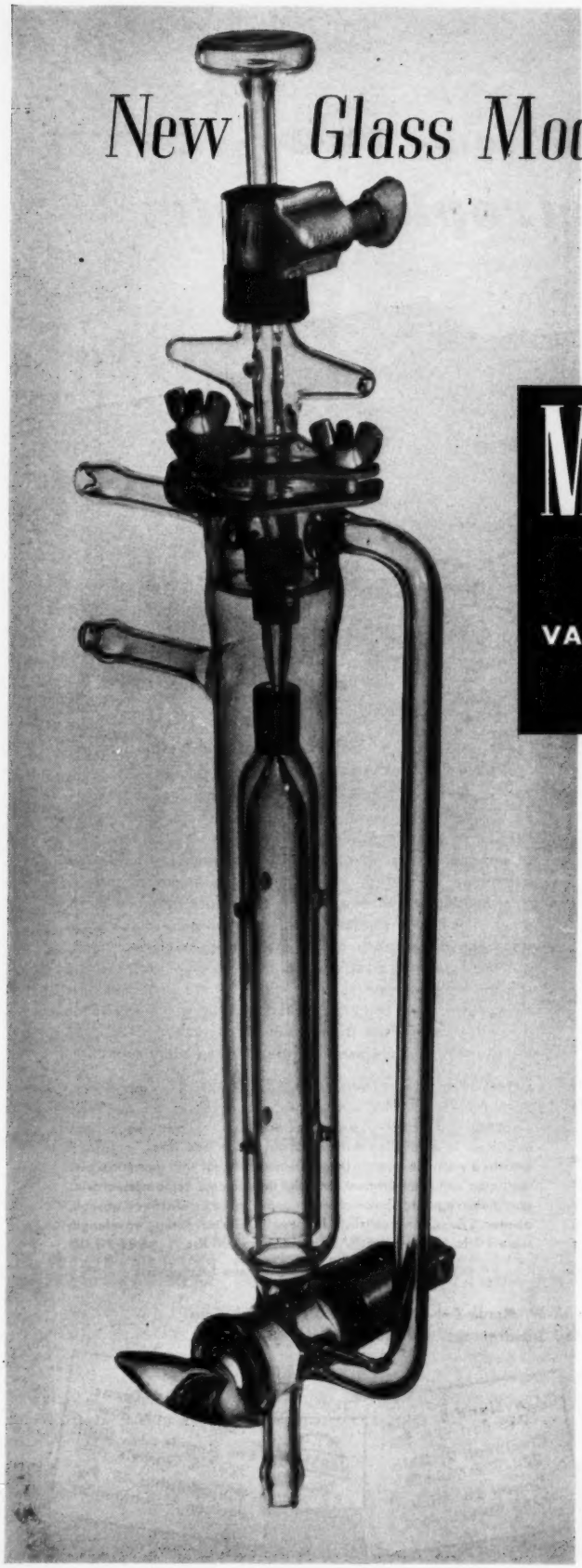

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*For Theory of Operation See Following Article:
Gilmont, R., Ind. Eng. Chem. Anal. Ed. *18*, 633, (1946); *23*, 157 (1951).

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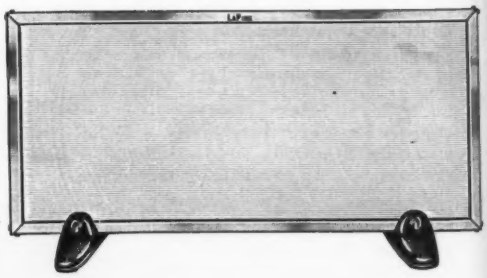

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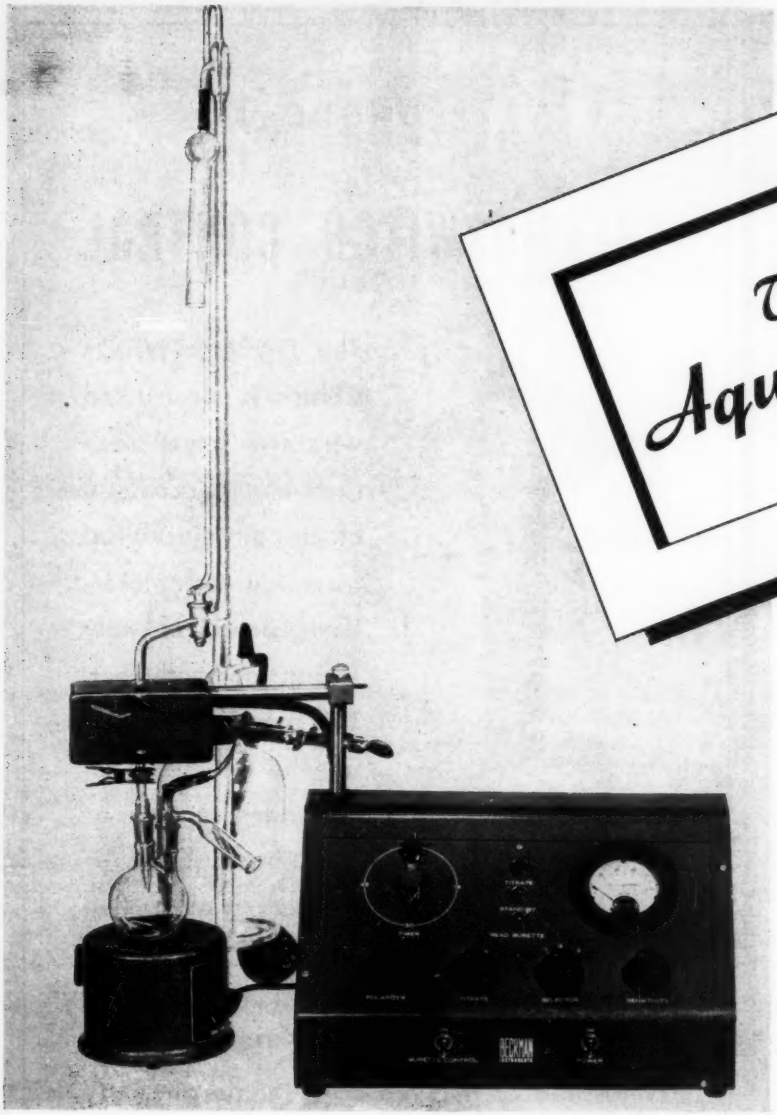

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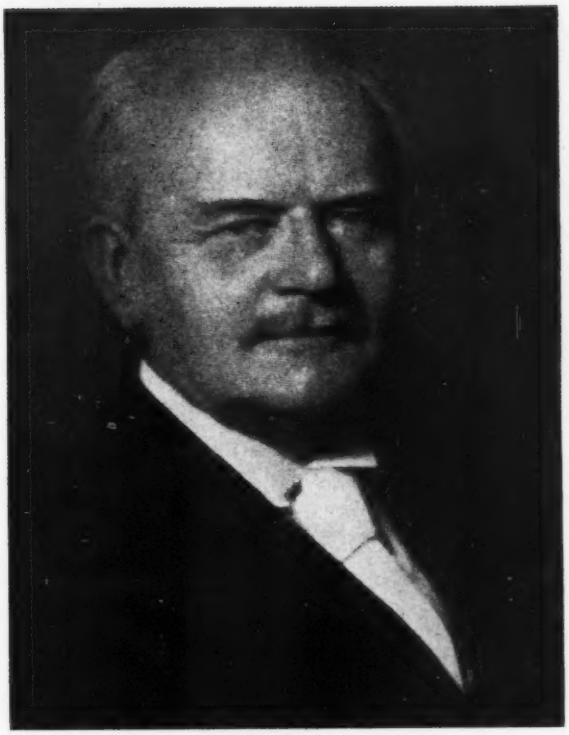

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(See page 76)

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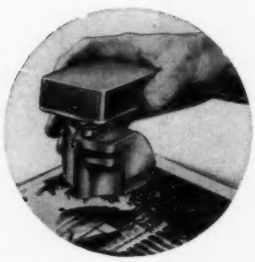

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Editor's Outlook

Not so very long ago Dr. Herman I. Schlesinger was given the Honor Scroll of the Chicago Chapter of the American Institute of Chemists, in well-earned recognition of his many years of service at the University of Chicago. Two remarks which he made on that occasion have stuck in my mind ever since I read his address of acceptance. They mark him as a man who thinks independently, if unconventionally, and is not content to "run with the pack."

> I am convinced that the principles and philosophy of science, distilled away from its phenomena and its techniques, are, like the brandy distilled away from the wine, a heady potion likely to produce a false glow of accomplishment rather than the strength resulting from solid intellectual nourishment.

We need to be continually reminded that science is a method of contemplating nature, and unless we *know* nature the contemplation is not only fruitless but perhaps even a sign of mental unbalance. We cannot theorize until we have something to theorize *about*. Perhaps the man who said "Sometimes I set and think and sometimes I jest set" wasn't so stupid; at least he had sense enough to save his mental energy until he had something to apply it to. It is proper and useful to point out that the mere collection of data is not science, that such a collection only becomes scientific when imagination is brought to play. On the other hand, imagination without a factual basis may be good poetry but not science. The currently fashionable discussions of the philosophy of science and the scientific method tend to overlook the dull process of assembling information and emphasize the more spectacular features, but observation is a primary step in the scientific process and all the rest is empty without the necessary antecedent.

> It is my conviction that one of the most important, even if an indirect consequence of scientific thinking, has been its contribution to the ideals in whose pursuit this country was founded, and in whose defense we have fought two world wars and are again arming ourselves. The ideals of liberty and freedom are the consequences of the concept of the dignity of the individual. In my opinion, this concept owes as much to science as it does to philosophy and to religion. It is to a great extent based on the contention of science that man is primarily a part of nature and not primarily a part of a political, a social, or an economic order. As a part of nature, each man's needs, and particularly his potentialities, should, in so far as is consistent with his responsibilities to his fellow men, be determined by natural laws, and by the endowments nature has given him rather than by man-made laws or man-made distinctions of caste, or of economic, political, or social stratification.

This is the answer of the pure scientist to the applied-scientific contention that all of man's efforts should be directed to making his social life happy, comfortable, and congenial. If there is any basis for contention between the natural scientist and the social scientist, this is it. In the specialized educational field it draws a line between "nature-centered" and "child-centered" education, between the conception of education on the one hand as a study of the natural environment of which man is a constituent part, and on the other as a preparation for the individual's daily life in a social group. As a statement of fact there is little doubt about it; as a basis of action it will not be universally agreed with—depending upon one's socio-political leanings, no doubt. Nevertheless, it is a point of view well worth considering.

"ALLOTROPIC" BROMINE

An Investigation of a Crystallized Red-Brown Sample of a Substance Originally Labeled as "Bromine—Pure, for Analytical Purposes"

EUGENE G. ROCHOW and IRA KUKIN
Harvard University, Cambridge, Massachusetts

ON OCTOBER 12, 1949, the following letter was received from Mr. D. W. Jayne, Jr.:

Dear Dr. Rochow:

Relating to the tube of "bromine" that I sent you in July, I have now received from Mr. Arnott (the gentleman who sent me the material) the history of this sample. I am sending this to you because I think you will be interested in his dissertation.

MEMORANDA re Sealed Tube of BROMINE from Laboratory of T. Davidson-Arnott 31st January '45

The following deals with a sealed glass tube, 4 3/4" long by 3/4" (external) diameter, formerly containing about 1 1/2 inches *liquid* BROMINE. At the end of what I believe to be nearly 50 years, the BROMINE, which I have never heard of as existing in crystalline form at temperatures higher than from 7° to 12° below zero F., remains *solid* in an ambient temperature of 84°F.

HISTORY. This is quite simple. Around 1893–4, not long after my arrival in Trinidad, I was engaged in research work on Crookes Tubes, following up the experiments of Lenard and afterwards Goldstein (canal rays). I had built and was using a 6"-Spark Ruhmkorff Coil. I blew and exhausted my tubes, roughing out with a 36 lb. Toepler and finishing with a five-fall Sprengel (I still have all of the old equipment); at a later date I replaced the Toepler by a Fleuss Oil Pump; (in 1898, for X-ray work, I built and was using a 15"-Spark Coil). After exhaustion I frequently introduced various gases, keeping the tubes on the pump for days, and sealing-off by a gasoline blowpipe. I also investigated the effect of cathode rays (through "windows") of alkali halides and minerals, but am in a position to state that at NO time was the BROMINE subjected to rays of *any* kind. When, in 1896, we received a description of the tube used by Roentgen, I was able to duplicate it, and the first X-ray photographs in Trinidad I made a little later in the same year.

Around, I believe, the latter part of 1896 I imported from the London house of Messrs John J. Griffin & Sons, a considerable number (120 odd) of reagents in glass-stoppered bottles for restocking my chemical cupboards, and amongst them was this tube of Bromine in liquid form. As the Bromine was never required, the tube, from that day till this—nearly fifty years—remained in the one chemical cupboard reserved for 8-oz. bottles. At first it probably stood upright in a glass jar along with other tubes of inert substances—Burroughs & Wellcome's Solids (Starch, Oxalic Acid, green and blue dyes) and the like—but for the last twenty years it has lain on its side in a glass trough (6" × 3" × 1/8" thick) half filled with cetaceum: usually though not always, covered by the trough's flat glass lid (6 1/4" × 3 1/4" wide × 3" deep pierced with two 3/16" holes about 3" apart).

At all times the Bromine was in a liquid condition with brown vapour filling the rest of the tube, and when I looked at it within the last twelve months it certainly presented the same appearance as on its arrival in 1896 or 7.

But a few weeks ago, when relabelling a number of stock bottles I came across the tube which, meanwhile, had changed out of all recognition: with NO trace of vapour or liquid, the glass of the tube was perfectly clear, and, inside piled up pretty equally at either end, were masses of dry sharp crystals, the crystals at one end being larger and better-formed.

When I had last seen the tube (possibly a year earlier) the ink on the paper label had faded and was illegible, but I knew this to be the word "Bromine" written with a fine pen in somewhat elongated script, and presumably in ink made of iron and galls. I now handed the tube to a chemist friend to see if he could restore the writing by sulphides or other reagents, or make out the letters under the mercury arc, but the soft surface of the paper seems to have been eroded and he had no success. Unfortunately, perhaps through shaking or handling or exposure to heat, when, a fortnight later, the tube was returned, the contents had undergone a change for the worse and now showed up as solid, liquid, and gas, and in this condition it has remained.

I have no theory to account for the structural alteration, nor can I suggest whether the change is the result of small forces over a long period or a large one acting quickly. It is true that in 1909 (and for some twenty years thereafter) a great deal of X-ray work was carried out in the room in which the Bromine was stored. (I still have twenty-three gas tubes 6" to 8" diam. on the racks, and though I used heavy glass and lead-lined shields around the tubes, I shall never know how I escaped X-ray dermatitis). But I cannot believe the Rays had anything to do with it, and in any case no such work has been done there in the last fifteen years. Small quantities of radio-active barium and potassium platinocyanides are kept in (W. M. weighing) bottles on the opposite side of the room, and in the same cupboard, but not on any near shelf, are 8 oz. bottles of Uranium Oxide, Nitrate, Fluoride, etc. I cannot believe that any of these could produce a cumulative effect resulting (as by the introduction of a catalyst) in the immediate rearrangement of the structure of the contents of a sealed glass tube.

This Bromine was supplied by Messrs. Griffin as "Pure, for Analytical Purposes"; might it be that it was not technically Pure, and that it contained a percentage of Carbon Tetrabromide? Or (as possible as its date of manufacture) free chlorine (as in some American Bromines), free iodine (as in German) or Cyanogen? Could any of these bring about structural alterations?

As far back as 1872, Baumhauer, in the *American Journal of Pharmacy*, ascribed the variations in the generally-quoted Specific Gravities of Bromine (2.99 to 3.14) to the presence of water in the specimens showing the higher figures; might it be that when first introduced into the tube the bromine contained residual traces of water, and that, over a long period of years this water was occluded by the walls of the tube, after which, a mere shake might have brought about crystallization? Could the slight but long-continued vapour pressure at 84°F. for ten months in each year have produced an effect of any kind?

At a later date I carried out a long series of experiments on COLD LIGHT, using the ordinary ELATERID, of which I usually kept from 50 to 100 at a time in a wire cage in the laboratory. The work was begun at the instance of Dr. Raphael Dubois of Paris and Marseille with whom I had corresponded for years:

Dr. Dubois was the foremost authority on the subject and had published his very comprehensive volume; "Les Elaterides Lumineux," as far back as 1886. During this period I operated high-frequency Oudin and Tesla coils giving sparks four and five feet long, and phosphorescence, luminescence & fluorescence called for high-voltage tubes, infra-red and ultra-violet light, and the use of radio-active salts. Yet I cannot connect any of this work (nor the apparatus involved) with any change in the characteristics of the contents of a sealed glass tube relegated to the lowest shelf of a closed cupboard with heavy glass doors, and in a very subdued light.

13th February '45

I might add that BROMINE CRYSTALS have been described as of "a dark leaden colour and with a lustre nearly metallic" (U. S. Disp. 18th ed.); that, no doubt, was correct for the crystals then observed at *a low temperature*, but scarcely applies to those in my tube. So far I have had no opportunity to examine them for refraction or angularity, nor with the microscope, spectroscope or polariscope—in each test allowing for the curved glass wall (which, in places, is not free from hair-like air-ducts produced at the original drawing of the tube but closed with the sealing of the ends).

It would appear that conditions within the tube are not yet stabilized as on some of the few days on which I have looked it over, the traces of gas and liquid, at other times definitely in evidence, are all but negligible. So, until we know more about the facts it will be safest, perhaps, to keep the tube away from bright lights, changes of temperature or position, and free from shocks.

In a further handwritten report signed by T. Davidson-Arnott on July 20, 1949, the information contained in the previous letter was repeated, plus some other information designed to throw more light on this curious phenomenon. The pertinent new information follows:

Examination of the *tube* shows that the surface is covered with long minute veins: the original tube from which it was drawn out had a number of air-bubbles. Here the hair-tubes open out into hollow cells not unlike the mercury bulbs of small clinical thermometers. The free ends of those hair tubes open directly into the large tube as they were filled with liquid bromine, which is presumably, now solid . . . . One small blister (half-way between a large convex and a large concave blister) has nearly vertical walls and a deep well . . . . The inside of the tube contains brown gas or is stained brown: there is no liquid at this date—20 July 1949 . . .

In all probability the solid bromine will be found to be a polymer but we know that other substances are, after long periods, allotropic-sulphur, for example . . . .

On the other hand I have a recollection that in the case of bromine minute traces of water have a profound effect on structure, and if the sealed specimen was a very dry one or has dried out, in time, anything might happen. *When on 31st January 1945 I first discovered the profound change in what had always been a liquid, the process of crystallization was not complete: today, 4½ years later there is no free liquid left . . . .*

Some preliminary experiments were tried with the sealed tube before opening it, and it appeared that there were at least two phases present. When part of the tube was immersed in warm water, a volatile liquid condensed on the upper walls and as this ran down it solidified to lemon-yellow crystals which melted at 46°. There was left a darker solid which contained some of the melted yellow crystals and did not have as sharp a melting point; it appeared to melt in the neighborhood of 77° and was almost black when melted. The colors could be explained on the basis of interhalogen compounds, but not the melting points. It appeared that the only thing to do was to open the tube and analyze for other halogens, even though this involved the risk of losing or altering the crystals if they should indeed be a polymer of bromine.

PRELIMINARY ANALYSIS

One could speculate at length about polymeric bromine, allotropic bromine, and iodine monobromide, but a direct analysis of the material was clearly in order. A plan of analysis was devised and the tube was prepared for opening. The appearance of the glass before and after having been cut indicated that it was of a very poor quality of soft glass, containing numerous air bubbles and the hair-like lengthwise ducts mentioned by Mr. Davidson-Arnott. The material was under a pressure no greater than its own vapor pressure. The reddish-brown vapor discolored the skin and left a burning and irritating sensation. The crystalline solid was not altered upon opening the tube.

Samples of the material were withdrawn and subjected to qualitative tests for bromine and other halogens. It was found that the substance dissolved in carbon disulfide and carbon tetrachloride to form a reddish-brown solution, and in chloroform or glycerol to give a yellow color. The material dissolved in an aqueous solution of potassium bromide to give a brown solution which distributed itself in two layers when carbon disulfide was added. The solution in potassium bromide gave no blue color with starch, indicating that no free iodine was present. The original material dissolved in a solution of potassium hydroxide to form a slightly yellowish solution which, after neutralization with acetic acid and reduction with sodium hydrosulfite, gave a slightly yellowish silver halide precipitate upon addition of a solution of silver nitrate. A small crystal of the original material when heated with concentrated sulfuric acid evolved a reddish-brown gas but no violet color appeared. Chlorine water was added to a solution of the material in carbon disulfide and a brown color only was produced in the carbon disulfide layer. A weaker solution of the material was reduced with a solution of sodium bisulfite and an excess of ferric alum was added. No iodine vapor was evolved upon heating. (This test was later repeated using zinc as the reducing agent.)

Since the presence of bromine (and no other halogens) was indicated by these tests, a small amount of the original crystalline material was added to a tube of pure liquid bromine to see whether it would seed the latter. The crystals dissolved, but to this date no physical transformation of the liquid bromine has occurred.

DETAILED ANALYSIS

Obviously the next thing was to test purity of the original sample by determining the percentage of bromine in it. While preliminary procedures were being worked out for this determination, a chance

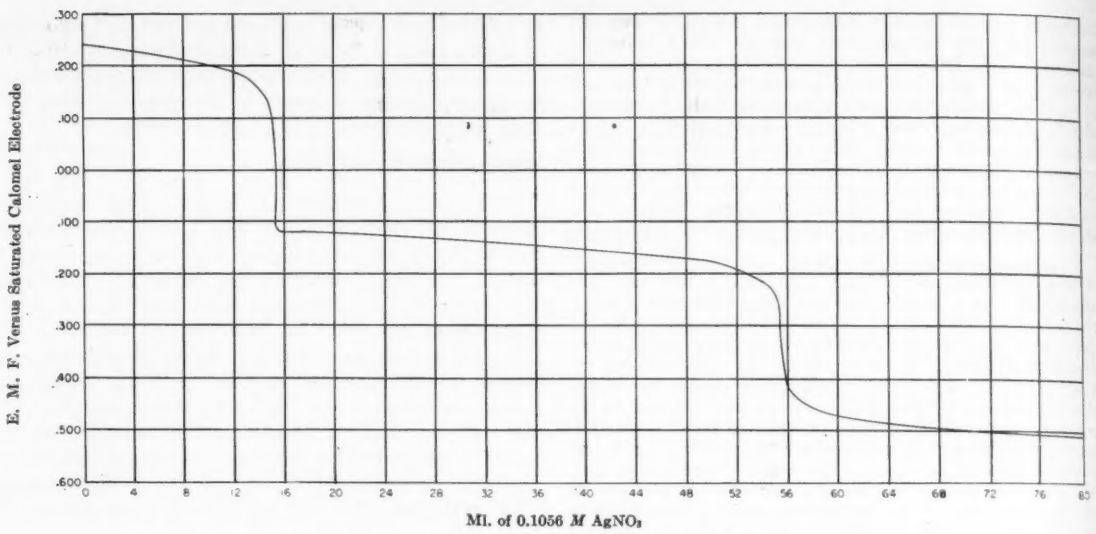


Figure 1. Amperometric Titration of Unkown Sample after Reduction with Zinc

observation cast doubt upon some of the previous conclusions.

The material dissolved in water to give a reddish-orange acidic solution. When neutralized with 12 *N* KOH, the solution passed through a brownish murky stage before becoming colorless after sufficient potassium hydroxide was added. Experiments using known bromine and iodine solutions showed that only in the case of the iodine solution was this intermediate brown stage present. Likewise, when the unknown was dissolved in a solution of potassium hydroxide to give an almost colorless solution and then was acidified, this brown intermediary stage appeared before the solution suddenly cleared at a definite pH. Comparable results appeared in the case of a known iodine solution, but not with a bromine solution. A reinvestigation for the presence of iodine in the original crystals was now undertaken. The unknown was added to a tube containing a mixture of chloroform, water, and 30-mesh granulated zinc. The chloroform layer now appeared violet and the water layer slighly brownish. Dilute acid was added and the reduction with zinc continued until both layers turned colorless.

The colorless solution then was heated in a Kjeldahl flask to which an acidified solution of ferric alum was added in excess. A pinkish vapor appeared which was condensed in a cold solution of potassium iodide. This gave a blue color with starch. It was concluded that iodine was present in the original sample, probably in the form of iodine monobromide (in which iodine has a positive oxidation state).

The above method was then made the basis for an exact determination of the iodine content. A weighed amount of sample was dissolved in water, reduced with 30-mesh zinc, filtered, and the filtrate acidified. Ferric alum was added in excess. The solution was heated in a Kjeldahl flask provided with a trap. The iodine was then distilled into a saturated solution of potassium iodide, which was cooled in an ice bath. This was then titrated with a solution of sodium thiosulfate which had been standardized both with recrystallized potassium dichromate and with a standard solution of ceric salt. Iodine was found to be present to the extent of about 53 per cent.

Another analysis to determine iodine and bromine simultaneously was then performed as follows: A 100-ml. volumetric flask containing 30 g. of potassium iodide and 15 ml. of water was weighed, the unknown was added, and the flask and contents weighed again. After diluting to volume, a 10-ml. aliquot was titrated with the standardized thiosulfate solution. The result was calculated as follows:

Wt. of sample = 1.4250 g.
Vol. of thiosulfate required for 10 ml. aliquot = 26.84 ml.
Therefore 26.84 ml. (0.0900 meq./ml.) = 2.416 meq. of I_2 produced per 10 ml. of sample.

If x = g. of I_2 present in an aliquot sample, $0.1425 - x$ = g. of Br_2 in aliquot sample and

$$\frac{x}{126.92} + \frac{0.1425 - x}{79.916} = 0.0024156$$

However, this equation gave a negative value for x. Only by assuming the presence of a halogen of lower molecular weight than bromine could this result have meaning. Could one, therefore, assume the presence of chlorine in the sample when all the previous tests showed such a preponderance of bromine and no chlorine?

A potentiometric titration was performed on the unknown which had been reduced with granulated zinc, using 0.1056 *M* $AgNO_3$. The results are shown in Figure 1.

The first break occurred at an e. m. f. of +0.010 v. (vs. saturated calomel) and the second break at +0.30

v.; the calculated percentages of halogens on the basis of iodide and bromide gave 52.89 per cent of iodine and 88.22 per cent of bromine. Moreover, the form of the above titration curve and the equivalent point potentials clearly indicated the presence of iodide with chloride and not iodide with bromide.

On repeating the previous experiment in which the reduced solution was oxidized with potassium persulfate (liberating any iodine and bromine), and then adding silver nitrate to the solution, a white precipitate was obtained which readily dissolved in ammonia water and reprecipitated on addition of nitric acid. Also, to the reduced solution from which iodine had been expelled with ferric alum, potassium chromate and concentrated sulfuric acid were added. Only the slightest trace of bromine was found.

A recalculation of the results of the potentiometric titration, assuming that the equivalence points were due to iodine and chlorine, gave 52.9 per cent of iodine and 39.1 per cent of chlorine. (In this titration small amounts of bromine would not show up and would be calculated as chlorine.) Moreover, a recalculation of the previous analysis in which the unknown was added to a solution of potassium iodide and titrated (assuming only chlorine and iodine to be present) gave 55.36 per cent of iodine and 44.64 per cent of chlorine. The material was clearly a mixture of iodine chlorides containing very little bromine.

In order to determine the exact proportions of iodine, bromine, and chlorine present in the sample, a gravimetric procedure based on a modification of the method of Bekk and Baubigney[1] was worked out. A weighed sample was dissolved in 0.5 *N* NaOH, acidified, and made up to a known volume. An aliquot portion was passed through a Jones Reductor containing zinc which had been washed with dilute sulfuric acid. The resulting halides then were precipitated with an excess of silver nitrate, washed, dried, and weighed as total silver salts. A second aliquot portion was treated in exactly the same manner, and the precipitated silver salts were digested with a solution of potassium dichromate in concentrated sulfuric acid. After all the chlorine and bromine had been expelled, the iodine remained in the form of iodic acid. The iodic acid then was reduced with a solution of sodium sulfite, and the resulting precipitate of silver iodide was washed, dried, and weighed. The filtrate (containing the silver formerly associated with the chlorine and bromine) was treated with a solution of potassium iodide and the precipitate weighed as AgI. This yielded:

Iodine	57.34%
Chlorine	39.96%
Bromine	3.64%
	100.94%

One cannot say definitely whether the bromine was present in the form of iodine monobromide, bromine chloride, or free bromine, but the presence of more than one phase seems highly probable in view of the following observations:

1. The solid begins to melt at 40° and continues until 70°, at which point a dark brown liquid forms; examination of the melting point tube reveals the presence of yellow stains along its sides.
2. If the sample is melted in a sealed-off tube, a dark brown solid forms at the bottom and yellow crystals appear along the sides as soon as the flame is removed.
3. If the sample tube is placed in an ice bath, fine needle-like amber crystals, yellow crystals, and brown patches appear on the sides of the tube besides the main mass of brown solid and a brownish oily layer. If a sample of the material is left in the refrigerator, after a few days cherry-orange crystals appear. Upon removal from the cold compartment, the orange crystals immediately turn reddish. When put back in the cold compartment, yellow-orange patches are formed.
4. If a sample of the material is added to liquid chlorine in a dry-ice–acetone bath to convert any iodine chloride present in the sample to iodine trichloride, the dark brown sample forms orange-yellow crystals which changed to a ruby color at room temperature, the color being noticeably different from that of the original sample. These crystals melt at 45–58° with decomposition. (The m. p. of ICl_3 is 47–62°, with decomposition.)[2]
5. If a crystal of iodine is warmed with a sample of the material and then cooled in a dry-ice bath, the reddish-brown crystals which separate change to a red-brown liquid at room temperature (m. p. ICl = 13.9° for brownish-red tablets of the β form, which is labile and changes into ruby red needles of the α form, m. p. 27.19°).

Summary of Analyses

	Volumetric titration of a KI solution of unknown	*Gravimetric*[a]	*Calculated, based on ICl*	*Calculated, based on ICl_3*
Iodine	55.36	57.34	78.16	54.39
Chlorine	44.64	39.96	21.84	45.61
Bromine	...	3.64	...	...

[a] To account for the discrepancies in the volumetric and gravimetric results, the following must be considered: The volumetric titration (in which the unknown was added to potassium iodide and titrated) is a simultaneous determination of two halogens, and in calculating the results the presence of the small amount of bromine was ignored. Also, each time that the tube was opened to withdraw a sample some vapor escaped. The composition of the vapor undoubtedly was different from that of the solid material left behind, due to the dissociation of iodine trichloride.

ABSORPTION SPECTRA

It might be concluded that the crystalline mass was a mixture of ICl and ICl_3, mixed with a small amount of IBr or BrCl. However, an analysis of the absorption spectra in the visible range might be expected to show exactly which interhalogen compounds were present.

Iodine monochloride behaves very much like iodine in that it changes its color depending upon whether it exists as a vapor, liquid, or solid. The color of the solid also depends upon the type of solvent in which it is dissolved, as shown in the table on the next page.

Samples of the original crystalline mass were dissolved in various solvents, and were found to form red-brown solutions in carbon tetrachloride, carbon disul-

[1] Bekk and Julius, *Chem.-Ztg.*, **39**, 405–6 (1915); *Chem. Abstracts*, **9**, 2042 (1915).

[2] Cornog and Kargas, "Inorganic Syntheses," McGraw-Hill Book Co., Inc., New York, Vol. 1, p. 165.

Solvents yielding[a]	*Solvents yielding*
(*a*) *yellow solutions with iodine chloride*	(*a*) *red-brown solutions with iodine chloride*
(*b*) *brown solutions with iodine*	(*b*) *violet solutions with iodine*
Methyl alcohol	Carbon tetrachloride
Ethyl alcohol	Chloroform (very pure)
Ether	Trichloroethylene
Ethyl acetate	Ethyl bromide
Formic acid	Chlorobenzene
Acetic acid	Hexane
Acetic anhydride	Cyclohexane
Hydrochloric acid	Benzene
Acetone	Toluene
Pyridine	Xylene
	Carbon disulfide

[a] GILLAM, A. E., AND R. A. MORTON, *Proc. Roy. Soc.*, **124**, 604 (1929).

fide, and benzene, and yellow solutions in methyl alcohol, ethyl acetate, hydrochloric acid, and chloroform containing a trace of alcohol.

The absorption maxima for the following halogens and interhalogen compounds dissolved in carbon tetrachloride are: chlorine, 332 mμ; bromine, 417 mμ; iodine, 520 mμ; bromine chloride, 380 mμ; iodine chloride, 464 mμ; iodine bromide, 490 mμ. Iodine trichloride has a strong absorption peak at 460 mμ and a weaker peak at 335 mμ (the only interhalogen compound to show such a double maximum). In alcohol and other solvating media, iodine forms a brown solution with an absorption maximum at 447 mμ. Iodine chloride and iodine trichloride form yellowish solutions with an absorption maximum at 335 mμ in alcohol while iodine bromide forms a yellow-orange solution with a maximum at 390 mμ in this solvent.

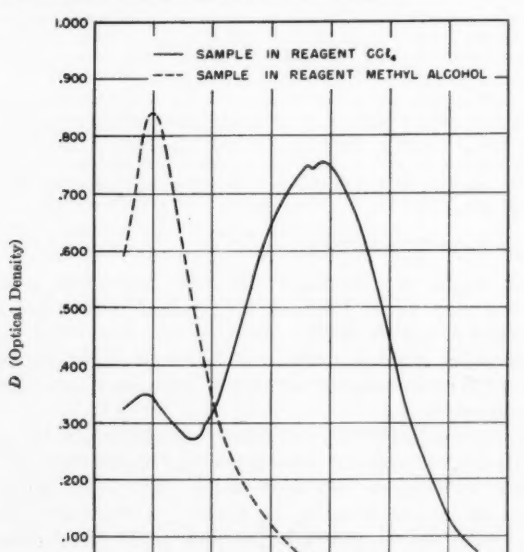


Figure 2. Absorption Spectra of Sample in CCl_4 and Methyl Alcohol

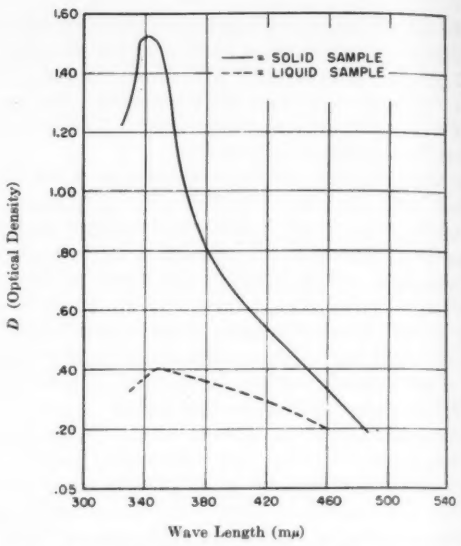


Figure 3. Absorption Spectra of Solid and Liquid Portions of Sample in $CHCl_3$ (Reagent)

The spectra obtained with samples of the "allotropic" bromine in two solvents are shown in Figure 2. The absorption peak in methyl alcohol at 338 mμ and the corresponding absorption in carbon tetrachloride at 458 mμ instantly rule out the possibility of free bromine, iodine, iodine bromide, or bromine chloride. Moreover, the second maximum in carbon tetrachloride at 335 mμ is unmistakable evidence of the presence of iodine trichloride. The single absorption peak in reagent chloroform is shown in Figure 3.

Figure 4 shows the changes which occur upon standing. The absorption in methyl alcohol decreases. In carbon tetrachloride, the maximum at 458 mμ decreases with a shift toward the shorter wave lengths, and the second maximum at 338 mμ (due to the chlorine) disappears with time. This can be traced to the instability of iodine trichloride and its liberation of chlorine as a result of dissociation, with the eventual formation of iodine chloride.

$$ICl_3 = ICl + Cl_2$$

This dissociation has been used in organic chemistry to get vicinal dichlorination.[3] Also, since the iodine trichloride spectrum is a composite of those of iodine chloride and chlorine, varying amounts of iodine chloride and iodine trichloride would give the same type of spectrum.

CONCLUSION

Only one further point requires consideration, and that is an explanation of how this sample (which originally was a liquid) solidified in a rather sudden fashion after the lapse of considerable time. The following facts must be borne in mind:

[3] CAMPAIGNE, E., AND W. THOMPSON, *J. Am. Chem. Soc.*, **72**, 629 (1950).

(1) Iodine monochloride when once melted can remain liquid below its melting point.

(2) Iodine trichloride exists as a solid below 40°C. and presumably the sample could not initially have been pure iodine trichloride.

(3) Bromine chloride is a very unstable compound, decomposing to bromine and chlorine.

(4) Iodine chloride has a large residual affinity for chlorine.

It therefore is proposed that the sample originally was a very impure bromine containing iodine and chlorine, allowing the possibility of formation of interhalogen compounds. As long as the bromine was present in excess, this sample would remain a liquid. With time most of the bromine which originally was present escaped, a likely possibility considering the shoddy condition of the glass in which it was contained and the presence of long capillary tubes in the glass, which were formed at the time the tubing was drawn and which probably were open to the interior instead of being sealed off as Mr. Davidson-Arnott had supposed. Any chlorine which would have formed due to the dissociation of bromine chloride would not escape but would react with the iodine chloride present, so that the concentration of iodine trichloride steadily increased. When enough of it had formed, the sample solidified.

To test this hypothesis, a small amount of the sample was dissolved in liquid bromine at room temperature and left at about 45°C. in a loosely stoppered test tube. After most of the bromine had escaped, the sample was cooled to room temperature. A dark-brown crystalline mass, very similar to the original solid, was left behind. While the above experiment is only suggestive, it is clear that all the evidence contradicts the existence of any "allotropic" form of bromine in the sample tested, and shows conclusively that the solid material is almost entirely a mixture of iodine chlorides.

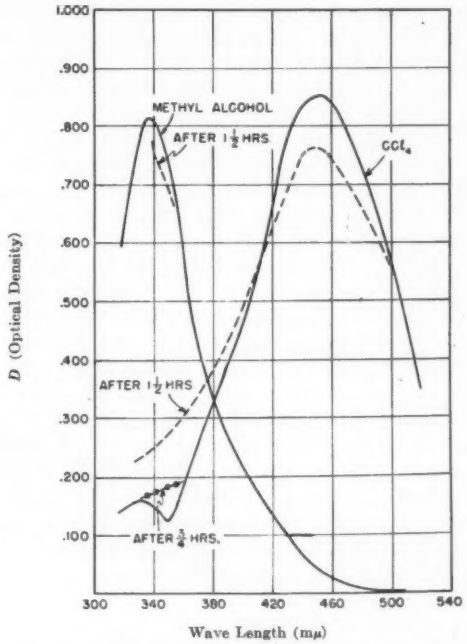


Figure 4. Absorption Spectra of Unknown in CCl_4 and Methyl Alcohol and Effect upon Standing

A STABLE RADIOACTIVE STANDARD

JOHN P. BUTLER and JOHN N. MANDAS[1]
Iowa State College, Ames, Iowa

DURING the course of radioactive investigations it was observed that ordinary black glazed porcelain ware exhibited residual activity. The source of such radioactivity was not at first readily apparent, but upon analysis it was found to lie in the hard black glaze as applied to such common laboratory utensils as evaporating dishes, crucibles, etc.

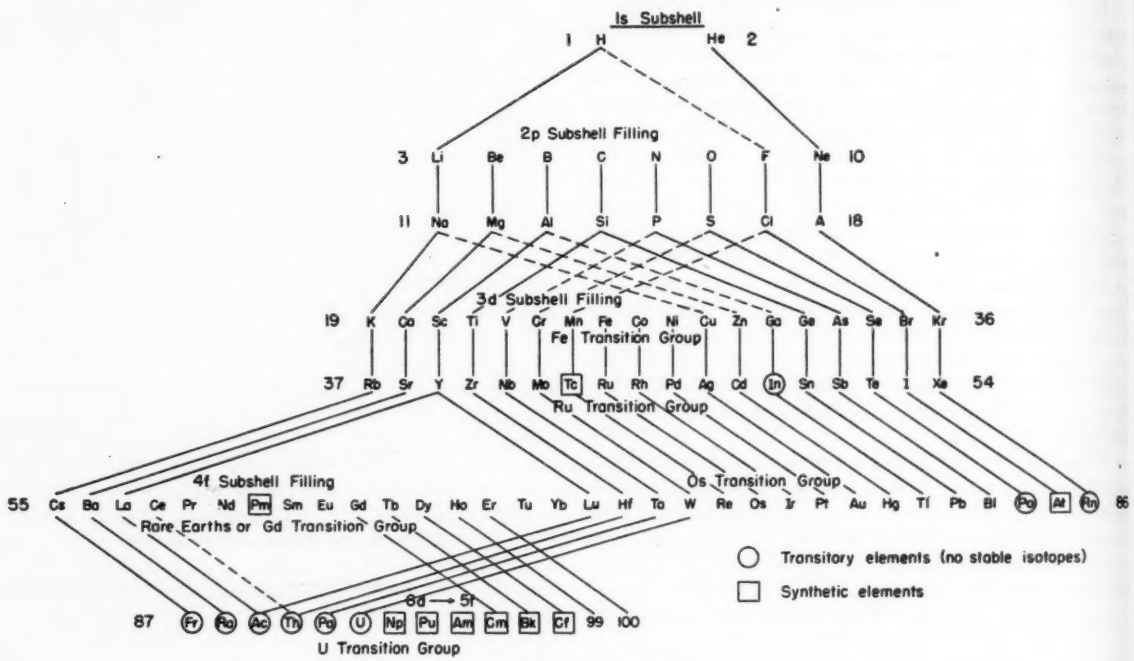
A communication from the Coors Porcelain Company, manufacturers of these items, revealed that the principal constituent used to impart this black glaze is uranium oxide of low percentage. The black oxide and other ingredients are suspended in water and the crucible or other piece dipped in the glazing mixture. The article is then fired at a temperature which vitrifies the body and melts the glaze leaving a hard, chemically resistant surface.

The relative chemical inertness of the procelain ware, its ready availability, and the long half-life of the radioactive constituents of the black glaze make the laboratory pieces suitable for standards of activity. The amount of radiation emitted depends on the geometry of the surface and other factors, but it is on the order of 2×10^{-3} microcuries/cm^{-2}. Possible uses include those of Geiger-Müller tube calibration, determination of tube geometry, and demonstration purposes.

[1] Present address: Western Cartridge Co., Alton, Illinois.

THE PERIODIC TABLE: THE *6d-5f* MIXED TRANSITION GROUP[1]

CHARLES D. CORYELL
Massachusetts Institute of Technology,
Cambridge, Massachusetts


1s Subshell
1 H
He 2
2p Subshell Filling
3 Li Be B C N O F Ne 10
11 Na Mg Al Si P S Cl A 18
3d Subshell Filling
19 K Co Sc Ti V Cr Mn Fe Co Ni Cu Zn Ga Ge As Se Br Kr 36
Fe Transition Group
37 Rb Sr Y Zr Nb Mo Tc Ru Rh Pd Ag Cd In Sn Sb Te I Xe 54
Ru Transition Group
4f Subshell Filling
Os Transition Group
55 Cs Ba La Ce Pr Nd Pm Sm Eu Gd Tb Dy Ho Er Tu Yb Lu Hf To W Re Os Ir Pt Au Hg Tl Pb Bi Po At Rn 86
Rare Earths or Gd Transition Group
Transitory elements (no stable isotopes)
Synthetic elements
6d → 5f
87 Fr Ra Ac Th Pa U Np Pu Am Cm Bk Cf 99 100
U Transition Group

Periodic Table in Long Form[2]

IN A recent paper in THIS JOURNAL, Glockler and Popov (*1*) have given succinctly the arguments for emphasis of the long form of the periodic table (or chart), usually attributed to Bohr (*2*).

Glockler and Popov propose the principle: "Since the periodic table should represent the variation of chemical properties as a function of the atomic number, it seems reasonable to expect that all the elements should be included in a single picture." They do, however, consider it necessary to make certain compromises, especially to present the transactinium elements in the framework of the Seaborg actinide hypothesis (*3*).

A little consideration will indicate that the conventional periodic chart has four principal features to act as vehicles for chemical information: (1) the breakdown into rows; (2) the use of columns or inter-row tie lines to emphasize close homology; (3) the use of broken tie lines to represent secondary analogy or relationships; and (4) the absence of tie lines coming down from above to indicate the incidence of an electronic transition group of a type not previously occurring in the table. With relatively few modifications, the Bohr-type periodic table presented by Glockler and Popov can be made to reflect more instructively the rather complex relationships obtained in the neighborhood of the *4f* or gadolinium transition group and, more importantly, in the *6d-5f* sequence extending from actinium ($Z = 89$) through the region of uranium and the synthetic earths to element 103, eka-lutetium. Such a periodic table was discussed in detail by the author in a parallel publication (*4*), and is presented here in the figure.

The breakdown into rows of the periodic table in the Glockler-Popov and the present variants is, as is conventional, based on the largest total quantum number n of the electrons in the valence shell, as indicated by the valence shell of the alkali and alkaline earths occurring as first and second members of each row beyond helium. The difference in this regard in the figure

[1] This work was supported in part by the Atomic Energy Commission.

[2] Reprinted here by courtesy of the *Record of Chemical Progress*, Wayne University, Detroit, Michigan.

from the Glockler-Popov form is only that the noble gases are not listed on the left edge in duplication of their position on the right edge of the previous rows. In other words, electronic structure....$(np)^6$ is not repeated as ....$(n+1)\,s)^0$ although this duplication is commonplace.[3]

One of the chief virtues of the frequent discussions of the optimal formulations for the periodic table is the one that has been dominant since the formulation of the table by Mendeleev (1869) and Meyer (1870): the discussions have high instructive value and the competitive proposals stimulate new experiments and greater appreciation[4] of apparently uncorrelated facts, as emphasized in the historical work of Mary E. Weeks (*5*). The purely chemical arguments about the transitions in electronic levels in the elements from actinium through californium have already been presented in detail (*4*). Suffice it to recall that in condensed systems the chemical properties in the sequence actinium, thorium, protactinium, and uranium are closely analogous to the properties in the sequence lanthanum or lutetium,[5] hafnium, tantalum, and wolfram (tungsten).[5] This has been interpreted (*4*) as a consequence of the predominately *6d* character of the valence electrons in the sequence, with any *5f* character not being energetically distinguishable. Thus the vertical tie lines in the Glockler-Popov form relating cerium, praseodymium, and neodymium with thorium, protactinium, and uranium should be replaced with the pre-1946 tie lines descending backwards from hafnium, tantalum, and wolfram.

The halt at oxidation number +6 in the highest known (sexipositive) oxidation state MO_2^{++} of the sequence neptunium, plutonium, and americium (*7*) indicates incontrovertibly that beginning with neptunium each element has successively one energetically more deep-lying electron than the previous one (*8*), the deep-lying electrons being certainly clear-cut *5f* representatives. There is no exact precedent for this in the periodic system, so that these elements show no close similarity with successive elements in any row above; they represent true uranides, with the same oxidation levels and ionic or molecular structures that are displayed by uranium. Vertical tie lines for these elements descending from promethium, samarium, and europium would thus be misleading, and should not be retained in a periodic table.

The presently available evidence (*3*, *4*) with regard to curium, berkelium, and californium indicates that homology with the *4f* transition group species gadolinium, terbium, and dysprosium is reasonably well established, the *5f* subshell energy levels having dropped appreciably below those of the *6d* level in aqueous solution and in solid compounds. The tie lines for these element-pairs presented by Glockler and Popov are thus acceptable and duplicated in the figure. Presumably the *5f* sequence will continue to its normal completion at element 103. Thus tie lines are shown for the next two elements most likely to be identified in modern nuclear-chemical element synthesis, elements 99 and 100[6].

The periodic table presented here shows fewer broken tie lines than does its Glockler-Popov analogue. Since broken tie lines represent secondary similarities, in the Von Antropoff manner (*9*), considerable variability is to be expected in their use, and limitations set by typography and clarity also affect their inclusion. There may be a case for a broken tie line between cerium and thorium; there is, however, a case against the use of one between europium and americium as shown by difficulties in preparing americious compounds (*4*), and the uranide analogies of the higher oxidation states.

Attention is paid in the figure to the absence of tie lines to emphasize the inception of new transition groups with the filling of subshells as indicated. In the first row[7] there is filled the $n = 1$ shell (called here the *1s* subshell since it consists of only the *1s* orbital); in the second row the series boron through fluorine is really a transition group associated with the filling of the three orbitals of the *2p* subshell, and in the fourth row from scandium through zinc there are filled the five orbitals of the *3d* subshell in a sequence often identified as the iron transition group. Only the third and fifth of the rows are without intrusion of a transition group not displayed in a previous row. On the basis of simple theory (*4*), the seventh row, starting with francium and radium, should also be without a transition group. The effect of initial intrusion of the *6d* transition makes the early part of this row (through uranium) tie in closely with the fifth row.

Because of this parallelism, virtually all pre-1946 periodic tables abandoned the *4f* or gadolinium transition group to a separate line at the bottom of the periodic table, connected to lanthanum by an asterisk. This set a precedent for the postwar proposals (*3*, *10*) that the sequence from actinium through element 103 be treated the same way. The conflict between the

[3] Glockler and Popov (*1*) have made the interesting proposal that the neutron $_0n^1$ be included as the "element" of atomic number 0. This proposal certainly deserves support in chemistry, but neutron belongs in a zero row before hydrogen and helium, placed above helium. This position would also emphasize the singular chemical properties predicted for the neutron. The chemical symbol for neutron would probably be the unique one: n.

[4] To the credit of the painstaking work of the classical chemists, it should be noted that the atomic numbers of all elements known in 1910 were deduced at that period from the known chemical properties. The work of Moseley (1913) served to verify the values and to provide a powerful new mechanism for the search for the then unknown elements 43, 61, 72, 75, 85, 87, and the elements beyond uranium.

[5] For a discussion of the proposals of the International Union of Chemistry for element nomenclature and spelling, see ref. (*6*).

[6] It seems to the author that Glockler and Popov have been too generous in putting in solid tie-lines between the pairs cadmium-ytterbium, indium-lutetium, tin-hafnium, antimony-tantalum, and tellurium-wolfram. Most of these might be acceptable as broken tie-lines reflecting less close analogy.

[7] In common chemical parlance the row hydrogen-helium is often not counted, and the row from lithium through neon is often called that of the first-row elements.

filling of the *6d* and the *5f* subshells, first clearly marked at neptunium (*4*, *7*) creates a situation without exact precedent, and makes useful the recourse to the Bohr long-form periodic table (with the full width of 32 members), to be able to show both *6d* homology up to uranium, and the clear *5f* homology beginning with curium. The set of closely similar elements neptunium, plutonium, and americium cannot be connected by strong ties to any preceding element sequence.[8]

The knowledge of 15 known elements in the table comes at least in part from radiochemical observations (*11*) and nine of these elements were prepared by modern nuclear transmutations. Thus it seems desirable to distinguish naturally occurring elements without stable isotopes (transitory elements[9]) and synthetic elements from the stable elements. The first class is distinguished by circles around the symbol, and the second class by squares. This sort of distinction does, to be sure, take over a typographical feature that could have been used for conveying information of a strictly classical chemical character, such as the distinction between metals and nonmetals.

There is one future problem that periodic-table devotees may yet have to face in this generation that will complicate tie lines to the last row. With the great legitimate interest in element synthesis and in view of the spectacular successes already obtained in the Berkeley laboratories of Professor G. T. Seaborg in going four elements beyond plutonium, modern nuclear chemistry may well provide us with synthetic forms of some elements beyond element 103. It seems very probable (*3*, *4*) that the elements from curium on through 103 are true *5f* transition groups. Element 103 should then be a close homologue of lutetium and thus a close analogue of actinium, although with a somewhat smaller radius. The *6d* transition group will then start anew, and element 104 will be another eka-hafnium closely similar to thorium. Thus element 104 might also be called dyo-thorium.[10] Elements 105 and 106 will be eka-tantalum or dyo-protactinium and eka-wolfram or dyo-uranium. But element 107 will not be a dyo-neptunium because of the *5f* intrusion in the same row, and the elements will go on as eka-rhenium (107), eka-osmium (108), etc., probably with good corresponding homology, to be represented by heavy vertical ties.

[8] It does not seem useful to tie them all to wolfram. It should also be noted that it is rather difficult to oxidize americium to the states AmO_2^+ and AmO_2^{++}, and that the state Am^{+4} is not available in aqueous solution (*4*, *7*). Americium is, energetically speaking, close to being a good analogue of curium, but still it does not show chemical analogy with europium.

[9] Three transitory elements, indium, thorium, and uranium, have one or two radioisotopes of half life (*12*) long compared to the age of the elements, and are thus found abundantly in nature and are known fully from standard chemical operations.

[10] Dyo-, two, from the Greek δυο. The classic periodic table prefixes *eka-* and *dwi-* are from the Sanskrit for one and two and refer to vertical homology. The Greek root is introduced here for nonadjacent horizontal analogy.

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EXHIBITION

An exhibition of printed works and manuscripts by Antoine Laurent Lavoisier (1743–94) will be held at The Grolier Club, 47 East 60th Street, New York 22, New York, from Tuesday, February 19, to Tuesday, March 18, 1952. A printed catalogue will be available there at that time.

THE EARLY DAYS OF CHEMISTRY AT THE UNIVERSITY OF WISCONSIN

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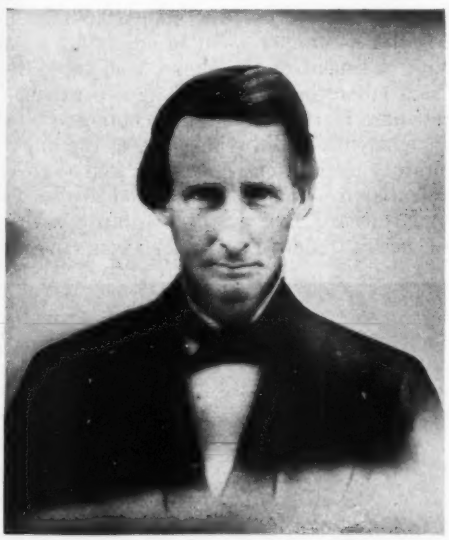
University of Wisconsin, Madison, Wisconsin

The first chair devoted exclusively to chemistry was created in the University of Wisconsin in 1880 when the institution was in its 32nd year. This does not mean, however, that the subject had finally received a belated recognition. W. W. Daniells, the first incumbent of the chair, had been with the University since 1868 as its first professor of agriculture and had been offering most of his instruction in chemistry. Even before the coming of Daniells, courses in chemistry had been offered by S. P. Lathrop and Ezra S. Carr.

Samuel Pearl Lathrop (1816–54), who had received his M.D. from Middlebury (Vt.) College in 1843, was appointed in 1854 to fill the chair of chemistry and natural history. This was the last of seven chairs to be filled in the institution which had been founded in 1848, the year that Wisconsin achieved statehood.



S. P. Lathrop about 1850

Lathrop, no kin to the University's first president, John H. Lathrop, had held a similar post since 1849 at Beloit College. Instruction in science was begun at the state university with the assistance of apparatus borrowed from the older college on the state border. Professor Lathrop, however, died before he had completed his first year.[1]

[1] Boutwell, P. L., *Trans. Wisconsin Acad. Sci.*, **41**, in press (1952).

Ezra S. Carr about 1865

Ezra Slocum Carr (1819–94), a graduate of Rensselaer Polytechnic Institute and Castleton (Vt.) Medical School, was successor to Professor Lathrop. Carr, a member of the faculty of Albany Medical College, was induced to come west not only by salary considerations but also by the fact that the Board of Regents strained the finances of the struggling University in order to purchase Carr's mineral collection for $1400.

Carr was favorably remembered by the naturalist, John Muir, as "The Doctor, who first laid before me the great book of Nature, and though I have taken so little from his hand, he has at least shown me where those mines of priceless knowledge lie and how to reach them."[2] In his teaching Carr called upon the students to repeat his lectures and demonstrations before the class. This method reflected the teaching procedure of Amos Eaton and his pupil James Hall, under whom Carr studied at Rensselaer. Though Carr referred often to laboratory work, these lecture demonstrations were the only opportunities offered to the students for experimental work. No systematic pattern of laboratory work was offered until Daniells appeared upon the scene.

Carr also busied himself with University politics, becoming a member of the Board of Regents in 1857.

[2] Bade, W. F., "The Life and Letters of John Muir," Houghton Mifflin Co., New York, 1924, Vol I, p. 143.

John E. Davies about 1895

Though he vacated the position a year later as a result of faculty opposition, he was a factor in the resignation of Chancellor Lathrop and an unsuccessful candidate for the vacant position. His tactless aggressiveness in promoting his department and deriding others made him unpopular with faculty colleagues, regents, and politicians. Nevertheless, he retained his professorship of chemistry and natural history for 12 years.

This period was a precarious one for the University and all those connected with it. Financial resources were never adequate. The second chancellor, Henry Barnard, was in such poor health during his two-year tenure that administrative matters fell largely on the shoulders of John Sterling, professor of mathematics, natural philosophy, and astronomy. With the chancellor's resignation in 1860 a reorganization was inaugurated. All professorships were declared vacant and provision was made for the restoration of only five. Immediately thereafter the Civil War depleted the student body to negligible proportions. Sterling and the small faculty struggled along as best they could.

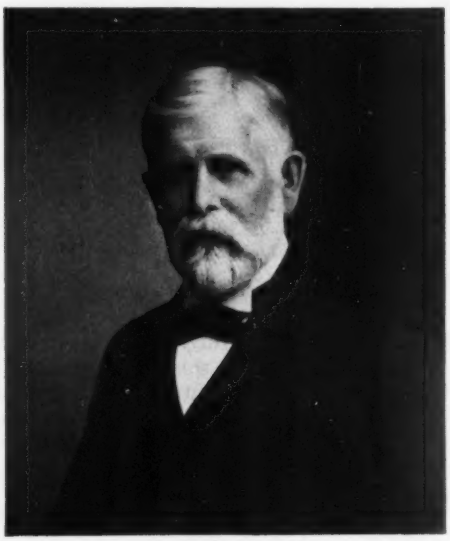
The University received a new lease on life with the onset of postwar days. Congress had passed the Morrill Act in 1862, setting aside forest lands to provide income for colleges of agriculture and mechanical arts. At first there was a serious consideration to the setting up at Ripon of a separate state college along the lines followed by Michigan, Ohio, Indiana, Iowa, and certain other states. In 1866, however, the state legislature passed enabling legislation for the incorporation of agriculture and engineering into the university.

Paul A. Chadbourne of Williams College assumed the presidency in 1867. He was responsible for adding John E. Davies and W. W. Daniells to the faculty in order to strengthen the sciences in accord with the new policy of emphasis on the practical arts. Carr was dropped from the faculty at this time. He was professor of agriculture at the newly founded University of California for several years before becoming Superintendent of Public Instruction in that state.

Dr. John Eugene Davies (1839–1900), a graduate of Lawrence University and Chicago Medical College, assumed the professorship of chemistry and natural history vacated by Dr. Carr. Davies' interests, however, lay more in the direction of physics and mathematics than in chemistry. His chemical responsibilities were terminated in 1874 when he became professor of physics and astronomy. A few years later, with the completion of Washburn Observatory, James C. Watson, the famous University of Michigan astronomer, was brought to the University. Davies spent the remainder of his life at the University as professor of physics, except for a short leave for study under Lord Kelvin. His published work was largely the result of his activities in charge of the Geodetic Survey in Wisconsin.

William Willard Daniells (1840–1911), a graduate of Michigan Agricultural College with an M.S. degree, came to Wisconsin in February of 1868 as the first professor of agriculture. Within the year, "and analytical chemistry" was added to his title. When Davies severed his connection with chemistry, Daniells' title was changed to Professor of Agriculture and Chemistry.

Laboratory instruction was inaugurated in the fall of 1868 after a summer spent in equipping a room in the south basement of University Hall (now Bascom, Hall) as a laboratory. Daniells had spent some time as a student at Harvard just before coming to Wisconsin and brought with him a realization of the importance of laboratory instruction which had been so effectively demonstrated by Wolcott Gibbs and Josiah Parsons Cooke of Harvard, and by the youthful Charles Eliot at the newly founded Massachusetts Institute of



W. W. Daniells about 1900

Technology. As early as the spring of 1868 Daniells had one student carrying on laboratory work on a carpenter's bench in the basement of University Hall. By 1875 laboratory instruction was being offered in qualitative, blowpipe, and quantitative analysis. There were also courses in organic and agricultural chemistry.

At the start of the fall term in 1880 Daniells severed his connection with the agricultural department and became the University's first professor of chemistry. Expansion began almost as soon as the department was created. At the time of his retirement in 1907 his department included three professors, eight instructors, and four assistants. In addition to expansion of personnel the physical plant had likewise expanded. The new department of 1880 shared a portion of the three-year-old Science Hall. When this building burned in 1884 it was replaced by the present Science Hall. The Regents decided, however, that such a noxious and dangerous subject as chemistry should not be taught in a fine building like Science Hall, so the chemistry department moved, in 1885, into its own Chemical Laboratory, the cream-colored brick building beside Lake Mendota which today houses the chemical engineers. The final move of the chemistry department was made late in the fall of 1905 when the central portion of the present Chemistry Building was completed.

Professor Daniells did not receive help in his teaching duties until 1879 when Charles Richard Van Hise (1857–1918) was added to the staff as assistant in metallurgy and chemistry. This young man was elevated to instructor a year later and became assistant professor of metallurgy and chemistry in 1883. He was not to remain long in the field of chemistry, however. In 1886 he became professor of metallurgy. Under the influence of President Thomas C. Chamberlin, he turned his attention more and more toward geological fields. He became, successively, professor of mineralogy and petrography, professor of Archean and applied geology, and in 1892, after he had won the first Ph.D. granted by the University, professor of geology. Only 11 years later he became president of his alma mater, the only graduate of the institution to achieve that distinction.

Organic chemistry was singled out for special recognition in 1885 when Homer Winthrop Hillyer (1859–1949) was made instructor in that subject. A native of Wisconsin, he had received a B.S. at the University in 1882 and then earned a Ph.D. at Johns Hopkins where Ira Remsen had built up the foremost research laboratory of organic chemistry in this country. Hillyer remained at Wisconsin for 20 years. When he left the academic world to become research chemist for General Chemical Company he was succeeded by William Frederick Koelker (1880–1911). The latter had just received his doctorate at the University of Berlin where he had been a student of Emil Fischer. Koelker's promising career was cut short by his untimely death in 1911.

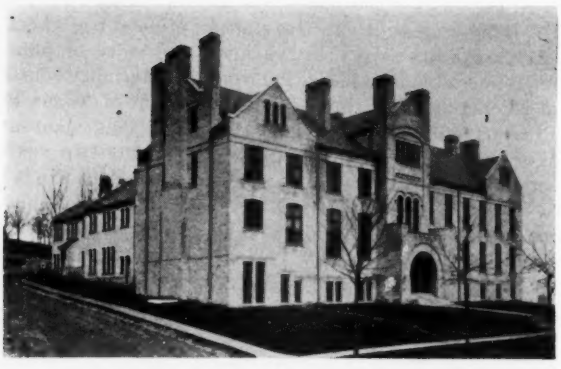
At the onset of Koelker's final illness, Richard Fischer (b. 1869) was added to the staff as professor of chemistry. Fischer arrived in chemistry by way of pharmacy. Following graduation from the pharmacy course at Michigan, where he had been a student of Albert B. Prescott, he had served as instructor in pharmacy at Wisconsin between 1894 and 1898. Taking two years for study abroad, he spent a semester with Emil Fischer at Berlin and then completed his doctorate in pharmaceutical chemistry under Ernst Schmidt, authority on alkaloids, at Marburg. He returned to Wisconsin as assistant professor of pharmacy, a position he held until he joined the chemistry department in 1909. During the years between 1903 and 1909 he also served as State Chemist, a position which had been held by Professor Daniells between 1879 and 1883.

Pharmacy also contributed to the inauguration of physical chemistry at Wisconsin by providing a place for Louis Kahlenberg (1870–1941) when he returned

Louis Kahlenberg about 1896

from Leipzig with his doctorate. Kahlenberg had shown such promise as an undergraduate at Wisconsin that he was made an instructor upon graduation in 1892. He earned his M.S. during the following year. His bachelor's and master's theses, carried out under the direction of Professor Hillyer, each resulted in a publication. He then felt the need for European study and gave up his instructorship. At the end of two years at Leipzig, where he studied physical chemistry with Ostwald, organic chemistry with Wislicenus, physics with Wiedemann, and botany with Pfeffer, he received the Ph.D. *summa cum laude*. His dissertation, done in Ostwald's laboratory, was an extension of his earlier studies on tartrate complexes with Hillyer.

Upon returning to Wisconsin, Kahlenberg was told



Chemical Laboratory. Home of the Chemistry Department 1885–1905. Headquarters of Chemical Engineering Department since 1905. Also Housed Physiology, Physiological Chemistry, and Pharmacology, 1907–17.

that the departmental budget did not permit his reappointment. A Canadian, Arthur P. Saunders, with a Ph.D. from Remsen's laboratory, had been engaged as instructor when he left. However, Professor Kremers, Director of the School of Pharmacy, was able to find funds to appoint Kahlenberg instructor in pharmaceutical technique and physical chemistry. The next year Saunders left the University to undertake further study in Europe and Kahlenberg became instructor in physical chemistry. Within two years he became an assistant professor and the year 1900 saw his promotion to a full professorship. Upon the retirement of Professor Daniells in 1907 he became head of the department.

The coming of Kahlenberg may be considered a milestone in the growth of the department. Being a man of strong character, he never hesitated to voice his beliefs, even when they were unorthodox. Though he left Ostwald's laboratory enthusiastic for the Arrhenius theory of electrolytic dissociation, he soon became critical of the whole concept. He inaugurated research in his own laboratory on the properties of solutions, particularly nonaqueous solutions. Students began graduate work at Wisconsin. Azariah T. Lincoln, who became professor of chemistry at Rensselaer Polytechnic Institute and later at Carleton College, received the first Wisconsin Ph.D. in chemistry in 1899. Herman Schlundt, who later became head of the department at the University of Missouri, received the second in 1901. Others followed and the reputation of Wisconsin as an institution for graduate study in chemistry began to grow. At the same time teaching was never slighted. Kahlenberg's self-confidence, his tricks of emphasis, and his showmanship caused his lectures to remain alive in the minds of students long after the courses and even the names of other professors were forgotten.[3]

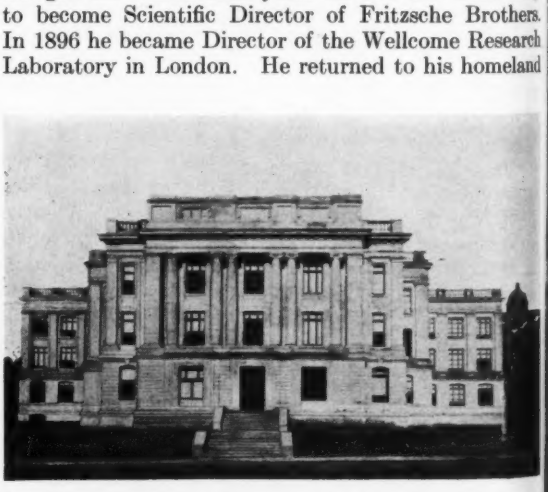
New men were added to the staff. Victor Lenher (1873–1927), who had received his Ph.D. under Edgar Fahs Smith at Pennsylvania, became assistant professor of general and theoretical chemistry in 1900. Francis Craig Krauskopf (1877–1947) was brought in as an instructor in February of 1906 on the recommendation of Wilder Bancroft of Cornell, under whom he was doing graduate work. Krauskopf mixed instruction with graduate work, earning his doctorate under Kahlenberg in 1909. James Henri Walton (1878–1947) was added to the general chemistry staff in 1907. He left a similar position at the University of Illinois which he had held since receiving his doctorate at Heidelberg under Georg Bredig in 1903. Joseph Howard Mathews (b. 1881), a graduate of Wisconsin who had served as an assistant in physical chemistry in 1904–05, was added to the staff upon completion of his Ph.D. under T. W. Richards at Harvard.

PHARMACEUTICAL CHEMISTRY

The growth of pharmacy paralleled the growth of chemistry and showed at all times a strong emphasis on the chemical aspects of the subject. The College of Pharmacy was founded by action of the Board of Regents in 1883.

President John Bascom, after consultation with his friend Ira Remsen, brought Frederick Belding Power (1853–1927) to Madison as professor of pharmacy and materia medica to head the department. Power had received a Ph.D. in 1880 at Strassburg where he had studied chemistry with Fittig and pharmacognosy with Flückiger. He had been teaching for three years at his undergraduate alma mater, the Philadelphia College of Pharmacy, before coming to Wisconsin.

Power was intensely interested in research and immediately inaugurated a program of student investigation. He himself was an active investigator of the essential oils and alkaloids although his greatest fame rests with his later studies on the constituents of chaulmoogra oil. After nine years he left the University to become Scientific Director of Fritzsche Brothers. In 1896 he became Director of the Wellcome Research Laboratory in London. He returned to his homeland



Chemistry Building in 1905. Home of Chemistry and Pharmacy Departments. Extensive Additions made in 1913, 1928, and 1939.

[3] HALL, N. F., *Trans. Wisconsin Acad. Sci.*, 39, 83–96 (1949); 40, Part 1, 173–83 (1950).

in 1916 to take charge of phytochemical research in the U. S. Department of Agriculture.

When Power left the University in 1892 to enter pharmaceutical industry, Edward Kremers (1865–1941) was made director of the school of pharmacy. Kremers had come to the University soon after the course in pharmacy was inaugurated and received his diploma of Graduate in Pharmacy in 1886. Feeling that the Ph.G. represented vocational training only, he remained at the University two years more in order to qualify for the more academic B.S. degree. There followed two years in Germany where he received the Ph.D. in 1890 at Göttingen. His graduate work was done under Otto Wallach, the outstanding terpene authority of the time. Upon returning to Wisconsin, Kremers spent a year as assistant in pharmacy and another as instructor before being elevated to a full professorship upon taking over Power's duties. What Kahlenberg did for chemistry, Kremers did for pharmacy. A four-year course leading to the B.S. degree was inaugurated. Graduate students began doing work in plant chemistry and the first Ph.D. was granted to Oswald Schreiner in 1902.[4]

AGRICULTURAL CHEMISTRY

Agricultural chemistry acquired the importance of departmental status in 1883 with the coming of Henry Prentiss Armsby. Agricultural aspects of chemistry had received recognition from the beginning of the University, however. Lathrop had acquired a loyal following among Wisconsin farmers during his professorship of chemistry and natural science at Beloit College. He had been a popular lecturer on scientific agriculture, a contributor to the *Wisconsin and Iowa Farmer*, and an officer of the Rock County Agricultural Society and Mechanics Institute. In addition to his college duties and his medical practice he found time for the cultivation of a small tract of land and exhibited prize-winning poultry and garden produce at the Rock County Fair. His interest in scientific agriculture was a factor in his call to the state university (ref. 1).

His successor, Carr, is known to have offered courses in agricultural chemistry. With the coming of Daniells there was a serious attempt to develop the agricultural offerings of the University. Organic chemistry, as offered in 1875, included the chemistry of germination, nutrition, vegetable growth, decomposition, fermentation, and saponification. Agricultural chemistry covered the composition of soils, manures, and crops, and the chemistry of the dairy. During this time Daniells also had charge of the University Farm.

The first serious agricultural research was inaugurated in 1880 when agriculture and chemistry were severed. A Norwegian immigrant who had just graduated from the course in mining and metallurgy, Magnus Swenson (1854–1936), was appointed instructor in chemistry. Although he may have assisted in the analytical laboratory his main role was that of research assistant to Daniells and the professor of agriculture, W. A. Henry. As a senior, Swenson had upset the residents of Madison with his thesis, "The chemical analysis of Madison well waters," revealing cesspool pollution of the drinking water used in 96 per cent of the homes. His research project with the agriculture department involved the extraction of sugar from sorghum cane. His extensive and systematic study received a $2500 award from the U. S. Department of Agriculture as the best thesis on sugar chemistry. At this same time he became *persona non grata* in Madison when a highly publicized demonstration of the centrifugal separation of sugar crystals failed of its purpose and sprayed dark sorghum molasses over legislators, regents, faculty, farmers, and other invited dignitaries. Swenson accepted a position with a Texas sugar refiner. During the next 20 years he attained a notable reputation for his development of sugar processing machinery as well as other chemical engineering achievements.[5]

Henry Prentiss Armsby (1853–1921), who came from the Connecticut Agricultural Experiment Station as professor of agricultural chemistry in 1883, had received his Ph.D. at Yale and followed this with study at Leipzig. He was at Wisconsin only four years when he was made director of the newly founded Pennsylvania Agricultural Experiment Station where he carried out his extensive experiments on the respiration of large farm animals. Stephen Moulton Babcock (1843–1931) filled the vacant position.

Stephen M. Babcock about 1897

Babcock had been an instructor in agricultural chemistry at Cornell for a year and chemist with the New York Agricultural Experiment Station at Geneva for six years before coming to Wisconsin. His doctorate had been earned under Hans Hübner at Göttingen in 1879 in the laboratory of the then aged Friedrich Wöhler. In addition to his professorship of agricultural chemistry, Babcock also doubled as chief chemist

[4] URDANG, G., *Trans. Wisconsin Acad. Sci.*, **37**, 111–35 (1945).

[5] HOUGEN, O., *Norwegian-American Studies and Records*, **10**, 152–75 (1938).

of the Wisconsin Agricultural Experiment Station and, after 1899, as assistant director of the station.

While Babcock is probably best known for his invention of the test for fat in milk, his ideas on animal feeding were of major importance. He doubted that energy and protein values were adequate in planning animal diets. When he put some of his ideas to test on two heifers loaned by Professor W. L. Carlyle of the animal husbandry department the experiment was terminated prematurely when one of the animals died and the other was hastily recalled by the badly disturbed Carlyle. Babcock's younger colleagues in agricultural chemistry were to bring such experiments to completion at a later time.

Babcock's only associate in the early days was Fritz Wilhelm Woll (1865–1922), a Norwegian who came to Wisconsin after receiving his B.S. at Royal Fredericks University at Christiania. He received an M.S. at Wisconsin in 1886 and was made assistant chemist of the experiment station when Babcock came to Wisconsin. Woll was made assistant professor of agricultural chemistry in 1893 and, after receipt of his Ph.D. in 1904, became associate and finally full professor before he left the department in 1913 to become professor of animal nutrition at the University of California.

Edwin Bret Hart (b. 1874) was brought to the department in 1906 when Babcock wished relief from some of his duties. Hart had been chemist at the experiment station at Geneva, New York, and had had graduate training at Marburg and Heidelberg under Kassel. William Edward Tottingham (1881–1944), whose specialty was plant chemistry, was brought from Geneva at the same time. Elmer Verner McCollum (b. 1879) took a Yale Ph.D. under Treat B. Johnson in 1906. After a year's further study under Lafayette B. Mendel, McCollum became an instructor at Wisconsin. Before leaving in 1917 to become head of the biochemistry department at the Johns Hopkins School of Hygiene and Public Health, McCollum had arrived at his concepts of fat- and water-soluble vitamins and had undertaken vitamin studies on small laboratory animals. Harry Steenbock (b. 1886) became a graduate assistant upon receiving his B.S. in 1908. William Harold Peterson (b. 1880) was made an assistant the following year when he came to Wisconsin with an A.M. from Columbia. Both men were made instructors in 1910 while they were still doing graduate work under Professor Hart. Both remained with the department after receiving their doctorate, Peterson specializing in fermentation biochemistry, Steenbock in fat-soluble vitamins.

In 1911 Hart, McCollum, and Steenbock, with George C. Humphrey of the animal husbandry department, published their classic study of the nutrition of cattle fed on rations consisting exclusively of parts of a single plant, a study which revealed that the suspicions of Babcock were indeed justified.

Babcock retired in 1913, but this did not mean that he ceased working or being an influence. Almost up to the time of his death in 1931 he was a regular visitor to his laboratory. His jovial nature was phenomenal and his friendly interest, coupled with helpful suggestions, was continued inspiration to his younger colleagues.

SOILS CHEMISTRY

The chemistry of soils received its start when President Thomas C. Chamberlin brought Franklin Hiram King (1848–1911) to the University as professor of agricultural physics in 1888. King had been a student of Chamberlin some years earlier at the state normal school at Whitewater. After additional study at Cornell, King had served as professor of natural science at the state normal school at River Falls before coming to the University. During his 13 years at the University he prosecuted a vigorous program of investigation into the application of science to farm problems. While much of his work was physical rather than chemical he published "The Soil" in 1895 and was developing the interest in soil fertility which was to receive recognition in later years. After he left the University he spent a turbulent three years with the Bureau of Soils in the U. S. Department of Agriculture, where he found himself in disagreement with the chemists and their theories on availability of fertilizer potassium. His connection with the Bureau was severed on account of publication difficulties. The travels in China which followed resulted in the well-received "Farmers of Forty Centuries," an account of Oriental methods of maintaining soil fertility.

Andrew Robinson Whitson (1870–1945) entered the department in 1899. As a Wisconsin undergraduate he had become interested in geology, and he followed Chamberlin to Chicago when that scientist resigned the University presidency to devote his whole time to geological teaching and research. At Chicago Whitson studied chemistry under Stieglitz, Alexander Smith, and Nef.

At Wisconsin, Whitson became full professor and chairman of the department in 1901 when King left. The designation "agricultural physics" was dropped in 1906 and the department of soils created to indicate the emphasis on soil science. Charles William Stoddart (b. 1877) was made instructor in 1904 and assistant professor two years later but left in 1910, a year after receiving his Ph.D., to become professor of agricultural chemistry and later dean at Penn State College. Emil Truog (b. 1884) became a graduate assistant after receiving his B.S. in 1909 and rose through the ranks to become head of the department upon the retirement of Whitson.

Whitson quickly inaugurated a program of studies on soil fertility. With the assistance of Alfred Vivian, a chemist with the Agricultural Experiment Station, he proceeded with an extensive soil survey. Vivian soon went to Ohio State University as professor of

agricultural chemistry and later dean of the college of agriculture, but the studies on the chemistry of soils continued.

PHYSIOLOGICAL CHEMISTRY

The University gave early thought to the development of a program in the field of medicine. In 1856 a department of medicine was created and staffed, Ezra Carr being named to the chair of chemistry and pharmacy. On account of finances and other difficulties the department failed to become operative. When Henry Barnard became chancellor in 1858 there was much discussion of the importance of instruction in the application of science to public health. David Boswell Reid (1805–63), a graduate of the University of Edinburgh, was appointed professor of physiology and hygiene and was also made director of a museum of practical science. Dr. Reid had come from the British Isles in 1855 with a reputation as a teacher of practical chemistry and author of several textbooks. He was also an expert on ventilation and had been active in the installation of an air-conditioning system in the houses of Parliament. In America he gave lectures on the progress of architecture in relation to ventilation and the preservation of health, under the sponsorship of the Smithsonian Institution and the Lowell Institute, before coming to the University in 1859.

Reid's tenure at the University was terminated with the reorganization which followed Barnard's resignation from the chancellorship. With the onset of the Civil War, Reid became an army surgeon with the Union forces. He lost his life in 1863 as a result of illness contracted in line of duty.

The scientific aspects of medicine received formal recognition during the next four decades only to the extent of setting up a premedical course. At the turn of the century the state began to recognize the need for increased facilities for medical training and a preclinical program was set up in the University. Complete training through the clinical years was not made available until after the completion of the Wisconsin General Hospital in 1924.

With the inauguration of preclinical work in 1906, a department of physiology and physiological chemistry was formed. Joseph Erlanger was brought from Johns Hopkins to head the department. He remained only until 1910 when he took a similar position at Washington University, St. Louis. Harold Cornelius Bradley (b. 1878) came to the campus in 1906 to take charge of instruction in physiological chemistry. Bradley had just received his Ph.D. at Yale under Lafayette Mendel. Along related lines was the work of Arthur Solomon Loevenhart (1878–1929), a Johns Hopkins M.D., who became professor of pharmacology and toxicology in 1908.

These departments were housed in the upper floors of the Chemical Engineering Building until 1917, when they were given space in Science Hall. Physiological chemistry was separated from the physiology department in 1921.[6]

CHEMICAL ENGINEERING

Chemical engineering started as a branch of electrical engineering. The head of that department, Professor Dugald Caleb Jackson, (1865–1951), was interested in developing the electrochemical phases of electrical engineering since electrochemical industries were undergoing rapid growth toward the end of the nineteenth century. Charles Frederick Burgess (1873–1945) was made instructor in electrical engineering upon graduating from the course in 1895 and entrusted with the development of applied electrochemistry.

By 1898 a four-year course in applied electrochemistry was offered, under the direction of Burgess in the department of electrical engineering. During the subsequent years the need for greater emphasis on applied chemistry began to be recognized. Magnus Swenson, now a prominent chemical engineer and industrialist, who served as a regent of the University 1905 to 1911, was foremost in encouraging the expansion of the offerings of the University in this field (ref. 5). In 1905 the regents authorized the inauguration of a course in chemical engineering. Burgess was promoted to a full professorship of applied electrochemistry and placed in charge of the courses in chemical engineering and applied electrochemistry which were now severed completely from the department of electrical engineering. In 1909 Burgess' title was changed to professor of chemical engineering. He severed his connection with the University in 1913, when his industrial activities were requiring too much of his time.

He had incorporated the Northern Chemical Engineering Laboratories in 1910 as a center for his consulting activities. The name was changed in 1915 to C. F. Burgess Laboratories. His work soon brought him into the dry cell business and he ultimately set up the Burgess Battery Company.[7]

The faculty of chemical engineering in 1905 consisted of Burgess, Assistant Professor Judson Dickerman, who handled the work in fuel technology and chemical manufacture, and Oliver W. Brown, who was instructor in applied electrical engineering and taught a course in illumination and photometry. The latter three men left the department before 1910.

Oliver Patterson Watts (b. 1865), who completed his Ph.D. in 1905, was made an instructor in chemical engineering the following year. He has remained with the department up to the present time and even

[6] OATWAY, W. H., JR., "Progress of the University of Wisconsin Medical School. The Years Before 1923," manuscript in the University of Wisconsin Medical School Library, undated.

[7] MCQUEEN, A., "A Romance of Research. The Life of Charles F. Burgess," The Instruments Publishing House, Pittsburgh, 1951.

after 14 years of retirement from teaching duties continues to carry on research in the electroplating of metals. Otto Louis Kowlake (b. 1878) became an assistant in the department in 1906 upon receiving his B.S. in applied electrochemistry. He became chairman of the department when Burgess left the University and left a long record of progress in the development of the department at the time of his retirement in 1948.

GENERAL TRENDS

This survey of chemistry at the University of Wisconsin, covering slightly more than a half century, marks the metamorphosis of a university. The original faculty, devoted to the broad cultural aspects of classical education, was somewhat oblivious to the wants of a vigorously growing state. Five years elapsed before the chair of natural science was filled. The first occupants were M.D.'s who offered not only chemistry but a spectrum of science offerings.

By the time of World War I the faculty was, in contrast, a body of specialists offering a wide assortment of courses to satisfy the apparent needs of an economy utilizing the findings of science. Men holding the Ph.D. degree were now commonplace and graduate instruction was developing vigorously. A university offering no instruction in chemistry during the first years of its existence had grown into one offering not only a complete listing of fundamental courses but having, in addition, five independent departments dealing with applied chemistry.

ACKNOWLEDGMENTS

The authors wish to express their gratitude to the numerous persons who provided information toward the development of this paper. Special thanks are due to the late E. A. Birge, H. C. Bradley, Richard Fischer, E. B. Hart, Olaf Hougen, the late Louis Kahlenberg, O. L. Kowalke, the late F. C. Krauskopf, the late E. Kremers, E. V. McCollum, J. H. Mathews, the late W. O. Richtmann, E. Troug, and George Urdang. Some of the material represented here was presented on Jan. 12, 1947, at a Phi Lambda Upsilon symposium on the "History of Chemistry at the University of Wisconsin," participated in by E. B. Hart, Olaf Hougen, H. A. Schuette, Emil Troug, and George Urdang. Some of the material was also presented by the authors at the Annual Meeting of the Wisconsin Academy of Sciences, Arts, and Letters in Milwaukee, Wisconsin, on April 11, 1947.

In addition to the references cited in the text, valuable material was obtained from biographical reference books such as "American Men of Science," University catalogues and other University publications, and the histories of the University by C. W. Butterfield (1879), R. G. Thwaites (1900), J. F. A. Pyre (1920), and M. Curti and V. Carstensen (1949).

IMPROVING VACUUM STOPCOCKS

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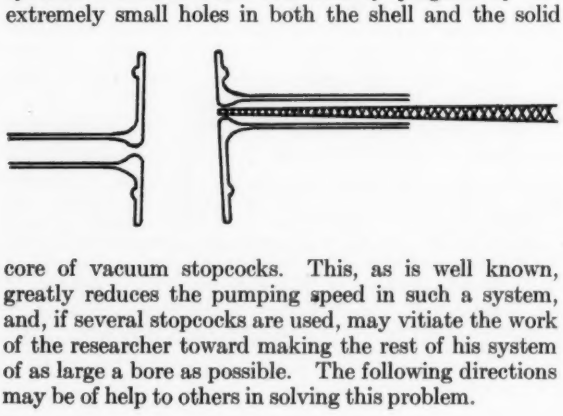
DURING the setting up of high-vacuum distillation systems we have been considerably plagued by the extremely small holes in both the shell and the solid



core of vacuum stopcocks. This, as is well known, greatly reduces the pumping speed in such a system, and, if several stopcocks are used, may vitiate the work of the researcher toward making the rest of his system of as large a bore as possible. The following directions may be of help to others in solving this problem.

Solid core, offset, vacuum stopcocks were used. The connecting tubing should lie on a straight line through the barrel as shown in the illustration. A small rattail file was moistened with a cutting compound composed of 62 g. of camphor and 90 ml. of turpentine in 45 ml. of ether and the holes in the barrel were carefully enlarged by twisting the file.

In order to enlarge the hole in the solid core, a brass rod from a set of cork borers was tapered at one end for a distance of about 2 in. The rod was placed in a low-speed drill press and the hole was drilled out using a slurry of number 120 carborundum and the aforementioned cutting compound as the grinding medium. The stopcock was held in the hand and drilling proceeded alternately on one side and then the other. As the drilling progressed care was taken that the rod did not bind in the hole. The drilling of each hole required about 20 minutes.

A CONSTANT-REFLUX-RATIO DISTILLING HEAD

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An improved constant-reflux-ratio distillation head has been designed which is especially suitable for analytical work involving the fractional distillation of mixtures which boil below 0°C. The head described herein has the following desirable characteristics:

(1) It can be used for both relatively high- and low-temperature distillations without the necessity of changing heads during the distillation.

(2) A constant reflux ratio is obtained regardless of the distillation rate. Almost any ratio can be obtained by designing the head properly, and the ratio can easily be changed during the course of a distillation.

(3) The head operates equally well under vacuum or atmospheric pressure.

(4) The head is adaptable to precise distillations involving either homogeneous or heterogeneous distillates.

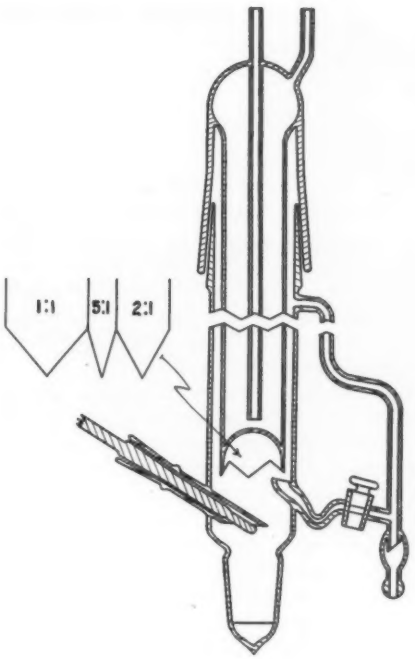
(5) The temperature of distillation is accurately measured.

(6) The head provides a cold distillate when low-boiling liquids are being distilled.

Of the numerous distillation heads recorded in the literature, the two which seemed most desirable for fractional distillation of mixtures boiling below 0°C. were described by Berg[1] and Lloyd and Hornbacher.[2] Both of these have most of the advantages of a constant reflux-ratio head; however, when used for lower temperature distillations the ground glass joints become cooled by the condensate and freeze, and thus a change in reflux ratio is prohibited during the distillation. The Berg head also suffers from the disadvantage that when used for vacuum distillations the safety vent at the top of the condenser must be connected by flexible tubing to the receiver, thus restricting the ease of rotation of the head when a change of reflux ratio is desired.

The distillation head illustrated in the figure employs a 28-mm. (o.d.) cold-finger condenser which is attached to the body of the head by means of a 40/50 ground glass joint. When the condenser coolant is acetone or other organic solvent at dry-ice temperatures, the dead space between the ground glass joint and the condenser provides adequate insulation for the former and keeps it from freezing solid during the course of the distillation. Any suitable stopcock grease may be used.

In operation, the vapor condenses on the convex side (outside) surface of the cold-finger condenser, as well as on the concave end of the condenser. This end is constructed so that the condensate which collects on the surface drains onto a number of tips. The size of the tip determines the reflux ratio. Any desired ratio may be obtained, and, if desired, vertical strips may be placed on the condenser tube in order to divide it into various surfaces. If vertical separator strips are not used, however, it is necessary to keep the condenser tube clean and smooth in order to prevent channeling. The enlarged section of the figure shows the circumference of the condenser end with a tip arrangement which will give reflux ratios of 1:1, 2:1, and 5:1 (amount of reflux: amount recovered).



The condensate from the appropriate tip is collected in a small cup which leads through a 2-mm. bore stopcock to a drop counter and thence to the receiver. To change the reflux ratio it is only necessary to rotate the condenser so that the tip which gives the desired reflux ratio is above the collecting cup.

The vent tube is located below the 40/50 ground glass joint and is connected directly to the receiver take-off tube. This gives structural support to the apparatus, and no additional connections are needed for vacuum operation.

[1] Berg, L., *Ind. Eng. Chem., Anal. Ed.*, **18**, 54 (1946).

[2] Lloyd, L. E., and H. G. Hornbacher, *Anal. Chem.*, **19**, 120 (1947).

The thermometer can be inserted, as shown, through a 10/30 ground glass joint. The shielded bulb method illustrated was considered most desirable, but other types of arrangement are equally possible, *e.g.*, an unprotected thermometer bulb, a thermocouple well, or a closed thermometer well.

A SIMPLE PREPARATION OF PRIMARY-STANDARD SODIUM CHLORIDE

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SOMETIMES a student wishes to standardize a supposedly determinate silver nitrate solution, either because he suspects an error in its preparation or because he wishes to secure an additional check on the correctness of his work. He is usually offered for this purpose a commercial reagent-grade, alkali-metal halide whose effective purity may be much less certainly known than that of the silver nitrate he used.

Most directions for the purification of these salts include several recrystallizations from water. This is a time-consuming process, and, in addition, it is inherently incapable of giving a good yield of highly purified product. An alternate procedure involves precipitation of sodium chloride from its cold concentrated solution with hydrogen chloride gas. This requires the construction of a hydrogen chloride generator, which is a nuisance. More important, however, is the fact that such a precipitation removes little, if any, of the potassium present in the original material. This paper describes a method for the purification of sodium chloride which gives a 70 to 80 per cent yield of material with an effective purity of 100.00 ± 0.01 per cent.

One kilogram of reagent or c. p. sodium chloride is dissolved in about 2.8 liters of water at 90–100°C., and the solution is slowly cooled to room temperature with occasional stirring. Ten ml. of concentrated hydrochloric acid is added, and the mixture is filtered through a medium-porosity, sintered glass funnel. The precipitated material, which contains the insoluble substances originally present, most of the potassium, and much of the bromide, is discarded. About 500 ml. of concentrated hydrochloric acid is added to the filtrate, which is then allowed to stand overnight, preferably with continuous stirring. The slurry is filtered as before and the solid is washed three times with 100-ml. portions of 4 *M* hydrochloric acid. It is thoroughly air-dried by suction, and is finally dried at 180°C. in an oven permitting free access of air for not less than 24 hours. If desired, the product may be dried further in an electric oven at 500°. Under these conditions, the loss in weight in 48 hours is close to 0.03 per cent, and seems to represent only water.

A number of samples of sodium chloride thus prepared were analyzed for chloride gravimetrically after drying at 500°. All of the precautions essential in very accurate work were observed, with the single exception that the solubility of the silver chloride in the mother liquor and the wash liquids was disregarded: this could hardly have caused an error greater than about 0.03 per cent. After drying at 300°, the weight of silver chloride was found, from the mean of eight analyses, to be 2.4522 ± 0.0003 times the weight of sodium chloride taken. The excellent agreement with the theoretical value of 2.4521 is partly due to the opposing effects of solubility errors and retention of water by the silver chloride. No differences could be detected between samples prepared from material sold by three manufacturers.

The product appeared to be entirely free from sulfate, calcium, magnesium, barium, bromide, and iodide according to the tests outlined by Kolthoff and Stenger.[1] Spectroscopic examination showed traces of lithium and zinc to be the only detectable metallic contaminants. Saturated solutions of several samples in freshly boiled distilled water of pH 6.6 had pH values between 6.3 and 6.7, and thus contained no appreciable amounts of residual hydrogen chloride. Similar solutions prepared from material dried at 180° had pH values near 5.7, resulting from the retention of a trace of hydrogen chloride. This corresponds to, at most, 10^{-4} mol per cent of hydrogen chloride, which is quite insignificant for nearly all purposes.

Analyses of material secured by fractional precipitation with hydrochloric acid gave 100.01, 99.99, 100.02, and 100.00 per cent for the effective purities of the fractions prepared by the successive addition of four 125-ml. portions of acid. There thus appears to be no tendency for impurities to collect in any one fraction of the precipitated material.

The original reagent-grade material corresponding to the product whose analyses were given in the last paragraph, and the material which precipitated on cooling, were analyzed and found to contain 99.83 and 99.45 per cent sodium chloride, respectively, after drying at 500°.

[1] KOLTHOFF, I. M., AND V. A. STENGER, "Volumetric Analysis," Interscience Publishers, Inc., New York, 1947, Vol. II, pp. 251–2.

DYNAMIC NUCLEAR REACTION MODELS

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At an annual open house of the University of Illinois at Chicago, the department of physical sciences exhibited to lay people two dynamic models illustrating nuclear chain reactions. Initially a unique mouse trap model illustrated controlled and uncontrolled multinuclear reactions while a model of an atomic pile portrayed the practical use of the controlled nuclear reaction.

The mouse trap model consists of several mouse traps so arranged and modified that the firing of one trap initiates the firing of one or two other traps, each of which is responsible for successive firings. The actual firing mechanism is a cork ball attached to the release levers of the traps; as the first trap is released, the released spring causes the lever and connected ball to swing back. It is this ball which sets off the second succession of traps. These secondary traps must be so interlocked as to achieve proximity of their releases—allowing both to be set off by the initiating trap. Incisions are consequently made on the wooden base of the traps; these incisions—of two types—are illustrated for 90° and 120° divergencies in Figure 1. The placement of these arrangements within the model is shown.

The multiplication effect increases through two, four, and eight traps—along eight separate paths. Only one of these eight is designed for continuance.

It is of value to examine the parallelism between the model and the actual mechanism of chain reactions. The spring simulates the energy of the splitting nuclei

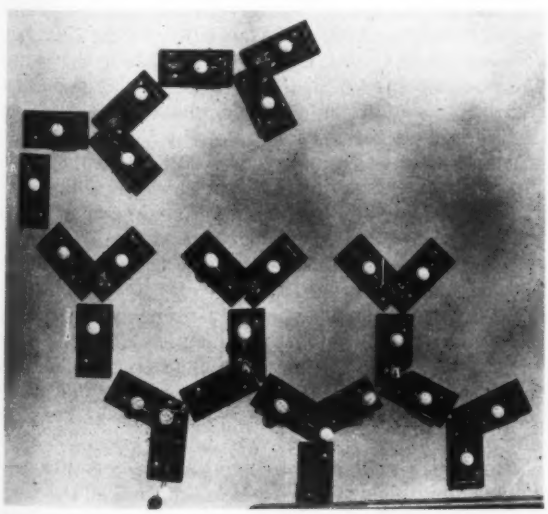

Figure 1. Mouse Trap Model of Nuclear Chain Fission Reaction

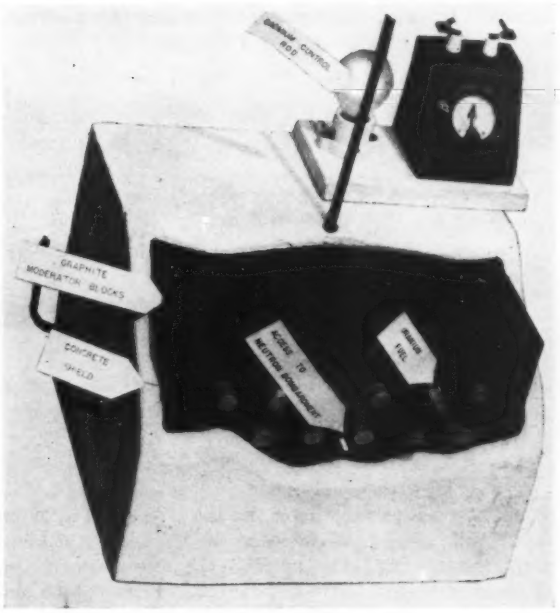

Figure 2. Model of Atomic Pile

and also imparts energy to the cork balls. These cork balls then in effect become fission neutrons. The absorption of neutrons is illustrated by the seven dead paths, and the continuing eighth path with its several dead subpaths as multiplication is averted.

The preparation of the traps and balls is evident from the figure. A flexible base to absorb shock (weatherboard), two bolts per trap, and appropriate paint will tend to increase the effectiveness and life of the model. An additional refinement of loosening the trap springs will result in diminished striking force of the trap, and wear on the cork balls, releases, and lever arms is greatly lessened.

This model is easier to construct than that of Slabaugh.[1] It also permits faster (4 minutes) and more frequent loading, and illustrates both branched and unbranched nuclear reaction chains.

The atomic pile is constructed from scrap lumber and standard electrical equipment.

A wooden cube, partially cut away, is the primary step of construction. This cube (a bottom face is unnecessary) is made to resemble a concrete shell by the application of a suitable stucco-like paint (Serotex Brand). An insert cube to this shell is made of scored

[1] Slabaugh, W. H., J. Chem. Educ., 25, 679 (1948).

(by sawing) and flat black painted stock to simulate the carbon moderator blocks. This insert needs no bottom or back faces. Sections of aluminum painted doweling are inserted into drilled depressions on the face of this inner box. At least one dowel should be lengthy and actually inserted into the model. A centrally placed section of small bore doweling represents the entrance for chemicals to be irradiated in the center of the pile. Glass tubing filled with colored liquid and attached through the concrete shield suggests the cooling and heat transfer system of the pile.

The general nature and importance of the cadmium control rods for real-life pile operation is illustrated by the following electrical feature. A ring stand steel rod, employed as the control rod, may be lowered or raised through the core of an assembly of choke coils, with the concomitant change of current impedance affecting the brightness of a glowing light bulb and the deflection of an ammeter needle. Wired in series with the choke coils the lowering of the rod reduces the intensity of the light bulb and meter reading, simulating neutron absorption and decrease of power of the pile.

BERTHOLD RASSOW

RALPH E. OESPER
University of Cincinnati, Cincinnati, Ohio

Professor Rassow, who at the age of 85 is still carrying on his full professorial duties at the University of Leipzig, forms a living bridge between the present and an era of chemistry that has now practically passed into history.

Berthold Leopold Peter Rassow was born at Bergen am Rhein on October 2, 1866. His father was a district judge, who in 1879 was transferred to Leipzig as a member of the Supreme Court. After passing the Abiturientenexamen, which qualified him to enter a university, Rassow served as a one-year volunteer in the infantry at Leipzig. He then turned to chemistry, and studied at Berlin and Leipzig. In January 1889 he was appointed private assistant to Johannes Wislicenus and then teaching assistant. The Ph.D. was conferred at Leipzig in 1890. For further training Dr. Rassow went to Munich, where he spent two years under A. Einhorn and Adolf von Baeyer. In 1892 he returned to Leipzig, where he habilitated as Privatdozent in 1895. In 1901 he was appointed Professor of Chemical Technology. When Wislicenus died (1902) and was succeeded by A. Hantzsch (1857–1935) in the fall of 1903, Professor Rassow became head of the newly created technological division. His life work had now begun, as he retained this post until 1939, even though he had reached the normal retiring age in 1935. Literally speaking, no adequate replacement could be found, either then or when the University of Leipzig resumed operations after World War II. One of the salient features of his teaching in technology has been the extensive system of works inspections in which he conducts groups of students over wide areas of Germany for periods of several weeks, and thus acquaints them with chemistry in large-scale action.

Professor Rassow's scientific publications have dealt largely with organic compounds, especially as they are related to dyes, dyeing, and textile chemistry, and to cellulose and lignin. Most of his many studies in this area served as doctorate dissertations, and have been published in the *Berichte*, the *Journal für praktische Chemie*, and the *Zeitschrift für angewandte Chemie*.

However, his activities have extended far beyond the walls of the laboratory. He was editor of the *Zeitschrift für angewandte Chemie* from 1904 to 1922; in this period the circulation as well as the scope of this important journal was approximately doubled. From 1907 to 1922 he was general secretary of the Verein deutscher Chemiker; he was equally successful in this capacity, but the many office duties did not appeal to him. He therefore decided to devote himself again principally to teaching, research, and writing.

He was scientific and executive secretary of the Gesellschaft deutscher Naturforscher und Ärzte from 1899 to 1938. He became particularly interested in the work of its educational committee, which later functioned as the Deutscher Ausschuss für mathematischen und naturwissenschaftlichen Unterricht. The movement for improving instruction in natural science and reintroduction of biology teaching was quite successful.

From 1912 to 1938 Professor Rassow, together with Paul Schmidt, and then with A. Lösche, edited the Wagner-Fischer *Jahresbericht der chemischer Technologie.*

From 1909 to 1938 he issued a series of "Technologischen Bilderbogen" designed for instruction in chemical technology. The well-known *Fortschritte der chemischen Technologie* (45 volumes) was under his editorship from 1922 on. From 1917 to 1939 he was managing editor of the *Journal für praktische chemie* and his name appears on the masthead of its successor, the *Journal für makromolekuläre Chemie*.

As a recognized authority, he has prepared reports on chemical matters in many trials and has acted as expert for the Reichspatentamt, the provincial courts of Leipzig, Dresden, Berlin, and the Supreme Court at Leipzig.

In 1931 he took over the very considerable task of revising Hermann Ost's classic "Lehrbuch der chemischen Technologie." The 18th to 21st editions of "Ost-Rassow" were issued under his guidance, and the 22nd edition is now in press.

His varied and extensive activities have made Professor Rassow one of the best known of the German chemists. His numerous friends in all parts of the world will be glad to learn that this lovable gentleman is hale and hearty and competently still meeting his many duties.

ELEMENTARY PHYSICAL CHEMISTRY AND MATHEMATICS

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ACCORDING to chemistry students, elementary physical chemistry is the most difficult subject encountered at the undergraduate level. From the teaching experience of himself and others, the author suggests certain main factors which contribute toward the frequent failures and consequent reputed difficulty which accompany an elementary physical chemistry course.

The essential cause of difficulty lies in the mathematical preparation of the students. Experienced physical chemistry instructors resign themselves to the necessity of training each new group of students not only in the appreciation of certain mathematical concepts but even in the use of some of the more simple tools of mathematics.

The more common mathematical difficulties are: (1) the use of logarithms, (2) the ability to recognize when integration is necessary, (3) the ability to recognize conditions under which integration is possible, and (4) the concept of the perfect differential. A few examples should suffice to indicate the general situation.

It appears that the teaching of logarithms by most mathematics instructors is concerned principally with methodology. The student is rarely required to express a logarithm in a usable form, especially in the case of numbers less than unity. To be sure, $\log_{10} 0.0045$ is $\bar{3}.653$, or even (heaven forbid!) $7.653 - 10$, but if the *numerical value* of the logarithm is desired both forms are useless. Why are students not taught to obtain the usable answer, -2.347, in the above instance? Then, of course, antilogues present the reverse of the same problem.

One simple instance will suffice to demonstrate points (2) and (3) above. Taking the expression $dw = pdv$, let us ask, when is it desirable to integrate such an expression and under what conditions is it possible to integrate? The student must be taught that it is necessary to integrate to obtain the total work along a prescribed path. The question of whether or not the expression can be integrated usually leaves the student in a quandary. At this stage in his education he should already know that integration is possible only if p is constant, or is an integrable function of v. This simple analysis of the equation, however, usually escapes the student whose calculus training has consisted of reproducing standard integrations with familiar variables. Such an approach on the part of calculus teachers and their textbooks has taken its toll of otherwise adequate students. The situation cited above is recurrent throughout physical chemistry.

As to the fourth and probably greatest difficulty of the physical chemistry student—the perfect differential or its counterpart, the line integral—one feels that the students have never before encountered the concept. Adequate mathematical preparation at this point would make their grasp of elementary thermodynamics almost automatic once the physical concepts were explained.

The teacher of physical chemistry should not have to teach elementary mathematics. We are in dire need of mathematics teachers who realize that mathematics is designed to be applied. Mathematics for its own sake always leaves the student wanting in his preparation for *anything* but more mathematics.

PROUT'S HYPOTHESIS[1]

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IT IS the purpose of this paper to focus on some of the less well-known aspects of the work of William Prout (1785–1850) and of the hypothesis that bears his name.[2]

THE ORIGINS OF PROUT'S HYPOTHESIS

When in 1816 Prout put forward the suggestion that all atomic weights were integral multiples of that of hydrogen, and that hydrogen may be considered the "protyle" of the ancients, the substance from which other elements are made, he remarked that the idea was "not altogether new."[3] In the literature of the history of chemistry there has been considerable discussion as to what Prout might have meant by this comment. Certainly Thomas Thomson was approaching the idea of integral atomic weights when in a paper in 1813 he noted that eight elements had atomic weights that were integral multiples of that of oxygen.[4] However it is probable that Prout was thinking of Sir Humphry Davy who, though not considering the question of relative atomic weights, had come very close to suggesting hydrogen as the primary matter. J. C. Gregory[5] and J. R. Partington[6] have briefly set out Davy's ideas but in the knowledge of this author Davy's views have never been quoted in detail.[7] They appear near the end of his "Elements of Chemical Philosophy"[8] published in 1812 both in London and in this country. The page numbers refer to the Philadelphia edition of 1812:

> We know nothing of the true elements belonging to nature; but as far as we can reason from the relations of the properties of matter, hydrogene is the substance which approaches nearest to what the elements may be supposed to be. It has energetic powers of combination, its parts are highly repulsive as to each other, and attractive of the particles of other matter; . . . [pp. 274–5].

His studies on ammonium amalgam suggested to him the composite nature of metals:

> On the most probable view of the nature of the amalgam from ammonia, as I have mentioned, it must be supposed to be composed of hydrogene, azote, and quicksilver; and it may be regarded as a kind of type of the composition of the metals; and by supposing them and the inflammable bodies different combinations of hydrogene with another principle as yet unknown in the separate form; all the phenomena may be easily accounted for, and will be found in harmony with the theory of definite proportions [p. 275].
>
> Even if it should be ultimately found that oxygene and hydrogene are the same matter in different states of electricity, or that two or three elements in different proportions constitute all bodies, the great doctrines of chemistry, the theory of definite proportions, . . . must remain immutable; . . . the numbers representing [the substances now considered as primary elements] . . . would probably be all found to be produced by the additions of multiples of some simple numbers or fractional parts [pp. 278–9].

The last part of this section must have suggested strongly to chemists that they analyze the characteristic weights of the elements to see whether simple mathematical relations could be found among them. It may well have been the stimulus that led Prout to his hypothesis of integral atomic weights. The passage is the more remarkable since Davy was only interested in combining weights, for he was doubtful, as we have seen, whether chemists had discovered as yet any true elements.

It is interesting that Prout himself doubted the validity of the whole atomic theory. In his Gulstonian lectures on the "Application of chemistry to physiology, pathology and practice" in 1831 he had suggested that the atomic theory was a "conventional artifice, exceedingly convenient for many purposes but [one] which does not represent nature."[9] He believed that the so-called "atomic weights" may merely be members of series of the type 3, 6, 9, 12, 15 and that other members of the series may be found in other chemical combinations of the substance. This view is criticized by Daubeny[10] who shows that Prout's view has the same consequences as Dalton's law of multiple proportions and is therefore an unnecessary complication. Prout defends himself in a communication at the end of Daubeny's book. It is here that Prout states his view that

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[1] Presented before the Division of the History of Chemistry at the 119th Meeting of the American Chemical Society, Boston, April, 1951.

[2] (*a*) An excellent paper by S. Glasstone, elucidating Prout's original papers and discussing Prout's hypothesis in its relation to the history of chemistry, appeared in J. CHEM. EDUC., **24**, 478–81 (1947). (*b*) Alembic Club Reprint 20, "Prout's Hypothesis," Alembic Club, Edinburgh, **1932**, contains not only the important papers by Prout, Stas, and Marignac but also a valuable historical introduction.

[3] *Ann. Phil.* **7**, 111 (1816). The paper is entitled "Correction of a Mistake in the Essay on the Relation between the Specific Gravities of Bodies in their Gaseous State and the Weights of their Atoms." The original "Essay" appeared the previous year, *ibid.*, **6**, 321 (1815).

[4] *Ibid.*, **2**, 114 (1813); see also ref. (2*a*).

[5] GREGORY, J. C., "A Short History of Atomism," A. & C. Black, Ltd., London, **1931**, pp. 142–3.

[6] *Chymia*, **1**, 120 (1948).

[7] Glasstone (ref. 2*a*) suggests that the views were never published, probably from reading Gregory. But it is clear from the agreement of Gregory's material with Davy's words that Gregory knew the original writings.

[8] DAVY, SIR H., "Elements of Chemical Philosophy," Bradford and Inskeep, Philadelphia, **1812**, Part 1, Vol. 1.

[9] DAUBENY, C., "An Introduction to the Atomic Theory," J. Murray, Oxford, **1831**, p. 62.

[10] *Ibid.*, p. 44.

perhaps the ultimate unit of matter is not hydrogen but some still smaller particle.[11]

Davy was intrigued by the possibility of a single primary matter manifesting itself in so many different forms:

> There is, however, no impossibility in the supposition that the same ponderable matter in different electrical states, or in different arrangements, may constitute substances chemically different [p. 278].
>
> [The idea of elements being] different arrangements of the same particles of matter . . . [has been] sanctioned by the authority of Hooke, Newton, and Boscovich. . . [p. 279].

Probably the greatest controversy has centered on the claim made by Professor J. L. G. Meinecke of Halle in 1816 that Dalton himself had anticipated Prout.[12] The question is discussed in detail in Alembic Club Reprint No. 20 (ref. 2*b*) in which the authors of the Introductory Section[13] come to the conclusion that Meinecke's claim had no basis in fact. Now Meinecke never explicitly suggested that the elements were constituted of hydrogen as the primary matter, nor did he claim that Dalton held this belief. He claimed that there was no need to refer to Prout when suggesting that atomic weights (called "Antheile" by him) were integral multiples of that of hydrogen since "according to Dalton, all stoichiometric magnitudes are multiples by whole numbers of the value of hydrogen."

Dalton believed that atoms were indivisible and he therefore could not conceive of hydrogen as the primary matter. But this does not dispose of the problem, for a type of Pythagorean number mysticism has often appeared in science and has at times received considerable confirmation from the strikingly simple mathematical laws that describe many natural phenomena. Considering the simplicity of the atomic theory and its power in correlating divers chemical phenomena, it would not have been difficult to believe that the numbers representing the relative weights of the atoms also obeyed simple laws.

Meinecke's claim receives strong confirmation from passages in Thomas Thomson's "History of Chemistry."[14] After quoting from his notes taken at a discussion with Dalton in 1804 that "the ultimate particles of all simple bodies are atoms incapable of further subdivision" he proceeds to discuss Dalton's atomic weight table of 1804 containing integral and half-integral values. A few pages later he comes to the 1808 table appearing in Dalton's "New System of Chemical Philosophy"[15] on which he comments:

> The following table shows the atomic weight of the simple bodies, as he at that time had determined them from the best analytical experiments that had been made. . . . He had made choice of hydrogen for unity, because it is the lightest of all bodies. *He was of opinion that the atomic weights of all other bodies are multiples of hydrogen;* and, accordingly, they are *all* expressed in whole numbers. He had raised the atomic weight of oxygen from 6.5 to 7, from a more careful examination of the experiments on the component parts of water. [Italics mine.]

It is almost inconceivable that this was an error of memory on Thomson's part. Thomson had published one of the earliest printed accounts of Dalton's atomic theory in the third edition of his "System of Chemistry."[16] As editor of the *Annals of Philosophy* he kept in close touch with chemical developments and wrote frequent reviews on the progress of chemistry. He was an ardent supporter of Prout's hypothesis and devoted much time to analytical studies which proved to his satisfaction the validity of Prout's view.[17]

It is strange that Thomson does not even mention Prout's hypothesis in his "History of Chemistry." He mentions the papers that led to the hypothesis only in connection with their significance in correlating volume relations and the atomic theory.[18] The reason for his silence is probably Berzelius' attack on Thomson's analytical work as being valueless.[19] In Berzelius' eyes Prout's hypothesis had no reliable evidence to support it. Thomson ends his "History" with a short explanation of his own work, admitting that some of it had not been too accurate. He continues to have the highest respect for Berzelius: "It is to his labours chiefly that the great progress which the atomic theory has made is owing."[20]

If Dalton did believe in integral atomic weights he did not believe in them for long, for in 1810 his table again contained some nonintegral values.[21] Taking Thomson's account as accurate, one must explain Dalton's references in 1808 to certain combining weights as being "nearly" whole numbers as a recognition of current experimental results being in disagreement with the whole-number theory. Thus: "Water is a binary compound of hydrogen and oxygen, and the relative weights of the two elementary atoms are as 1:7, nearly."[22] This would agree with Meinecke's statement that Dalton believed in integral atomic weights "without, however, calculating these values accurately by the aid of available analyses."[23] Meinecke flattered himself as having found integral atomic weights by the most careful investigation of all available analyses. This would exonerate Meinecke somewhat for making

[11] *Ibid.*, p. 129; this passage is quoted in ref. (2*a*).

[12] *Ann. Physik*, new series, **24**, 162 (1816).

[13] KENDALL, J., *Proc. Roy. Soc. Edinburgh*, **A, 63,** 1 (1949–50), reveals that he and L. Dobbin were the authors of this section.

[14] THOMSON, T., "History of Chemistry," H. Coburn and R. Bentley, London, **1831,** Vol. 2, pp. 289–94. Also *ibid.*, 2nd ed., **1833.**

[15] DALTON, J., "New System of Chemical Philosophy," R. Bickerstaff, London, **1808,** Vol. 1, Part 1, p. 219.

[16] THOMSON, T., "System of Chemistry," 3rd ed., G. Bell and Sons, Edinburgh, **1807,** pp. 424–9, 451–2. The section is reprinted in Alembic Club Reprint No. 2, "Foundations of the Atomic Theory," University of Chicago Press, Chicago, **1911,** p. 42.

[17] THOMSON, T., "An Attempt to Establish the First Principles of Chemistry by Experiment," Baldwin, Cradock and Joy, London, **1825.**

[18] Ref. (14), Vol. 2, p. 300.

[19] *Jahresber.*, **6,** 77 (1827); quoted in ref. (2*a*).

[20] Ref. (14), Vol. 2, p. 305.

[21] DALTON, J., *op. cit.*, **1810,** Vol. 1, Part 2, p. 560.

[22] *Ibid.*, **1808,** Vol. 1, Part 1, p. 215.

[23] *J. Chem. Physik*, **27,** 46 (1819). Translated in ref. (2*b*), p. 11.

use of Prout's and Thomson's values without mentioning them, since he clearly states that he used all available data.

PROUT AS EXPERIMENTER

Since Prout's hypothesis was enunciated on the basis of very incomplete data, and since later analyses showed the idea of integral atomic weights to be untenable as first formulated, it is common to think of Prout as a man of great speculative ability but of little experimental skill. Prout may have helped to foster this prejudice by prefacing his first paper (ref. 3) with a disclaimer as to his experimental ability (ref. 2*a*).

This view of Prout can be partly countered by pointing to his scientific achievements, including the discovery of hydrochloric acid in the stomach,[24] of uric acid in boa constrictor excrement (1815) and of murexide (1818), and his development of urine analysis and organic combustion analysis (1815–27).[25]

Perhaps even greater confirmation of his scientific ability can be obtained from the references to Prout's work in the writings of other scientists of his time.

When F. Wöhler performed his classical synthesis of urea he required the most authoritative analysis of natural urea in order to prove the identity of the two products. Of the available reports of analyses he chose Prout's as probably the most accurate.[26] When in 1830 Berzelius reported on Wöhler's synthesis he too used Prout's data in discussing the results.[27]

Edward Turner, who in 1829 was delegated by the British Association for the Advancement of Science to inquire into and report on the question of Prout's hypothesis, mentions that Prout had remarked to him the evolution of hydrogen chloride when fusing even the most carefully purified silver chloride. This was confirmed by Turner.[28]

But perhaps the most decisive testimony comes from Berzelius in a rather amusing connection. In his annual report on chemical progress for 1834 he states that Prout had reported at a recent Oxford Scientific Conference a difference in the specific gravity of air before and during the cholera outbreak in London. Then follows the significant comment: "From a less able observer than Prout, I would not have considered it worth while even to mention this result here."[29] This is high praise indeed when compared with his scathing attack on Thomas Thomson some years earlier as an investigator from whom science can derive no advantage whatever (ref. 19).

[24] *Ann. Phil.*, **24**, 117 (1824).

[25] Ref. 2*a*, and PARTINGTON, J. R., "A Short History of Chemistry," The Macmillan Co., New York, **1937**, p. 213.

[26] *Ann. Physik Chem.*, [2], **12**, 88 (1828). The passage appears in translation in WHETHAM, W. C. DAMPIER, "Cambridge Readings in the Literature of Science," Cambridge University Press, Cambridge, **1924**, p. 215.

[27] *Jahresber.*, **9**, 266 (1830).

[28] *Rept. Brit. Assoc. Advance. Sci.*, **1833**, 400.

[29] *Jahresber.*, **13**, 52 (1834). "Von einem weniger guten Beobachter als Prout, würde ich es nicht für der Mühe werth gehalten haben dieses Resultat hier nur zu nennen."

MODIFICATIONS TO FIT NONINTEGRAL ATOMIC WEIGHTS

The growing weight of evidence throughout the nineteenth century that atomic weights relative to hydrogen were not integral by no means saw the end of Prout's hypothesis as a fertile source for speculation.

I shall by-pass in this account the better known suggestions that the fundamental mass unit may be a particle smaller than hydrogen, and also Crookes' brilliant anticipation of isotopes. It was C. Marignac in 1860 who first tried to account for deviations from integral or half-integral values by suggesting a modification of the law of gravitation.[30] He saw Prout's hypothesis as a limiting law similar to the ideal gas laws, with deviations caused by complicating factors. His suggestion was that the cause that determined particular groupings of the primordial matter to form our atoms, also "exercised an influence on the manner according to which these groups of primordial atoms would obey the law of universal attraction, in such wise that the weight of each group might not be exactly the sum of the weights of the primordial atoms composing it."

A suggestion for the reason for expecting such a modification appears in a book by George Rudorf, a student of Sir William Ramsay, entitled "The Periodic System, Its History and Significance for Chemical Theory."[31]

> We know that the atoms mutually attract each other in the molecule; if an atom is a complex of "protyles," then we must assume that the protyles also attract each other in the atom. The force of cohesion between the protyles is very large compared with their weight; therefore it may—and probably does—influence the weight of a system of protyles. With molar quantities this attraction is extremely small; therefore, considering our ignorance of the nature of gravitation we have no right to assume that the weight, as we measure it, of n protyles is n times as great as that of one protyle. For it is likely that the Newtonian laws, which apply to molar quantities, do not hold rigidly when we deal with molecules; this has been pointed out by Pearson.

These speculations found their fulfillment in the discovery of the packing effect.

Lothar Meyer put forward a different solution. Commenting on Marignac's speculations he suggests the admixture of a ponderable ether to the primordial matter:[32]

> It is conceivable that the atoms of all or of many elements are essentially composed of smaller elementary particles of one primordial matter, perhaps of hydrogen, but that their weights do not appear as rational multiples of each other because in addition to the primordial particles, greater or smaller amounts of . . . luminiferous ether, which may not be devoid of all weight, may enter into the composition of atoms.

[30] *Bibliothèque Universelle* (Archives), **9**, 97 (1860). For the relevant portions of Marignac's paper see ref. (2*b*).

[31] RUDORF, G., "Das periodische System, seine Geschichte und Bedeutung für die chemische Systematik," translated from the English, L. Voss, Hamburg and Leipzig, **1904**, p. 271.

[32] Translated from MEYER, LOTHAR, "Die modernen Theorien der Chemie," 6th ed., Maruschke and Berendt, Breslau, **1896**, Book 1, p. 124.

Meyer's idea was incorporated into W. Preyer's "Genetic System of the Chemical Elements." In the introduction he states his hope that:[33]

> . . . the hypothesis of the derivation of the elements of high atomic weight from those of lower atomic weight and finally from one extremely finely divided primordial matter, or from hydrogen, or from hydrogen and the universally distributed ether . . . may soon . . . claim the rank of a theory.

This theory died with the modern recasting of physics which has discarded the ether concept, but the foregoing suggestions show the tenacity with which an idea is held even when disproved in its original form. Since Prout's hypothesis has been resurrected in the form of our knowledge of isotopes, it looks as though one should be as cautious about discarding a plausible theory as in putting forward a new one.

[33] Translated from PREYER, W., "Das genetische System der chemischen Elemente," R. Friedländer und Sohn, Berlin, 1893, p. iv.

A PERIODIC TABLE SHOWING THE RELATIVE SIZES OF ELEMENTS AND THEIR IONS

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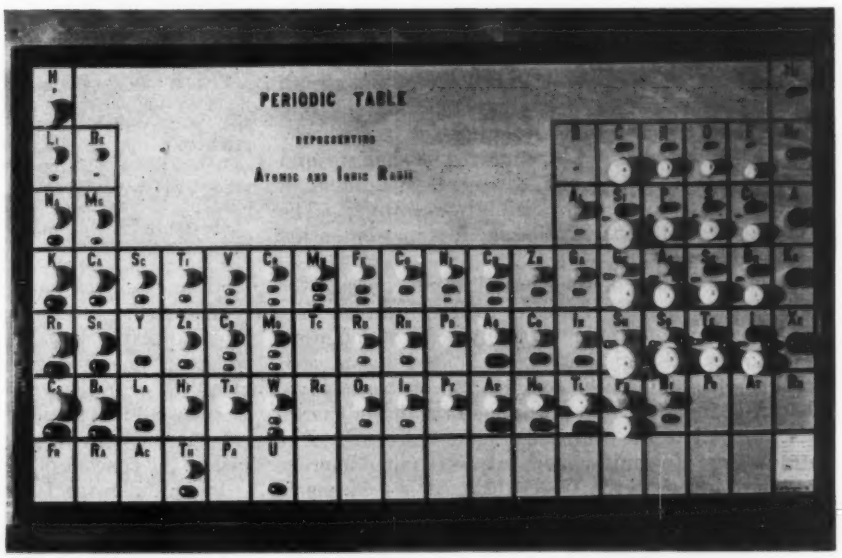

THE useful and instructive correlations which can be drawn between the sizes of atoms and ions and their physical and chemical properties have been ably presented by Campbell and others.[1–4] These generalizations are especially useful in teaching the periodic relationships in introductory chemistry courses because they are simple, logical, and readily visualized. At least one textbook has a section in which the influence of atomic size on periodic behavior is presented.[5]

A periodic chart representing the relative sizes of atoms and ions is a desirable teaching aid but is not readily available. Consequently the chart represented in the figure was constructed by a student at this university as a senior thesis project.

The following indicates the details of construction and cost of the chart. A 4 by 6 ft. plywood board $1/4$ in. thick is painted with 19 $1/4$-in. black vertical stripes $2^3/4$ in. apart. Eight horizontal stripes are painted in similarly, each separated by 4 in. The stripes are most conveniently painted by use of masking tape. Portions of the stripes are omitted to conform to the contour of the modern periodic table. This can be readily seen in the figure. The whole board is coated with a clear varnish and trimmed so as to leave a small margin beyond the outermost stripes. This is to accommodate a 1-in. molding which gives the board strength and rigidity.

A scale of 1 cm. equal to 1 Å. was adopted to calculate the sizes desired for the wooden balls. Balls representing atoms were painted white for metals, black for nonmetals, and gray for amphoteric elements; balls representing ions were painted black for cations and white for anions. All were fastened to the board by screws from the rear.

[1] CAMPBELL, J. A., J. CHEM. EDUC., **23**, 525 (1946).
[2] CAMPBELL, J. A., *ibid.*, **25**, 559 (1948).
[3] GARRETT, A. B., *ibid.*, **25**, 544 (1948).
[4] KELLER, R. N., *ibid.*, **28**, 312 (1951).
[5] MACK, E., A. B. GARRETT, J. F. HASKINS, AND F. H. VERHOEK, "Textbook of Chemistry," Ginn and Co., Boston, 1949.

THE CONCEPT OF RESONANCE ENERGY IN ELEMENTARY ORGANIC CHEMISTRY[1]

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THE teacher of a first-year organic chemistry course, who desires to impart a knowledge of the elements of the electronic theory of organic chemistry to his students, is faced with the problem of doing so in a way which offers some scope to their reasoning ability rather than requiring them to memorize an accumulation of data. Since elementary organic chemistry is generally taught in the second or third year of college, the question is how to present the electronic theory nondogmatically on a level adequate for sophomores or juniors.

There would be no problem in teaching electronic theory if we could rely on quantum mechanics, but we cannot take it for granted that the average first-year organic chemistry student has quantum mechanics sufficiently at his command. Therefore we inevitably have to make certain dogmatic statements and ask our students to take our word for them and accept and absorb them like experimental facts. The question is: shall we go to extremes and treat the entire electronic theory as something that has to be memorized, or shall we, and can we, try to keep dogmatic statements to a minimum and derive as many facts as possible by reasoning. The latter course is obviously desirable; it is my contention that it is also possible.

Resonance is one of the more difficult concepts for the beginner in organic chemistry to absorb, and in view of what was just said of his acquaintance with quantum mechanics it will help him little to be told that a wave function which is a linear combination of two or more other wave functions may correspond to a lower energy level than any of the component wave functions. But no knowledge of quantum mechanics is required in order to understand that the C—C distance in benzene is intermediate between the single bond distance and the double bond distance for carbon. That all C—C bonds in benzene are identical is simply a matter of deduction from the chemical properties of benzene, particularly from the absence of ortho isomerism. It is likewise no strain on understanding to learn that amides have acidic properties while amines are basic, or that chlorine in vinyl chloride or chlorobenzene is something very different from chlorine in ethyl chloride, and that the molecules of amides, of vinyl chloride, of benzene, and of many other substances can be best described by a simultaneous combination of two or more structures rather than by a unique structure.

This empirical approach to resonance is quite accessible even to beginners and gives some scope to their reasoning ability. It is therefore quite properly stressed by all textbooks for first-year organic chemistry.

But it is a different story when it comes to the concept of resonance energy. This is simply stated as a fact to be memorized. Let me quote from a few standard textbooks:

LOWY, HARROW, AND APFELBAUM (*1*): "The possibility of several electronic arrangements for the same atomic layout is connected with increased stability."

CONANT AND BLATT (*2*): "Whenever there is a possibility of resonance between two electronic structures . . . the compound is more stable than it would otherwise be."

FUSON AND SNYDER (*3*): "Resonating molecules are always more stable than would be expected from an examination of their structural formulas as ordinarily written."

FIESER AND FIESER (*4*): "A . . . consequence of resonance is dissipation of energy, or thermodynamic stabilization."

WERTHEIM (*5*): "The molecule is more stable than would be expected."

WILLIAMS AND HATCH (*6*): "The true structure may be regarded as . . . a resonating hybrid which is more stable than either."

MULDOON (*7*): "Compounds that resonate are characterized by stability."

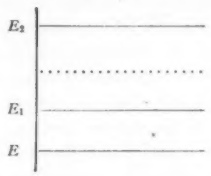
More examples could be quoted but the foregoing is representative of the dogmatic statements found in almost all textbooks. Those texts which do not present resonance energy dogmatically either do not present it at all (*8*) or use the concept without explaining it (*9*). Yet it would be possible to derive the entire concept of resonance energy by reasoning from experimental data, just as the concept of resonance can be so derived.

In the following I would like to give a presentation of how I attempt to go about arriving with my students at all the facts concerning resonance energy by a process of reasoning which is perfectly within reach of beginners of organic chemistry.

We base our discussion on two fundamentals. One is the chemical approach to resonance which defines resonance in terms of intermediate structures as an expression of experimental results; the other is the concept of energy levels and the greater stability of a lower energy level. To chemists and chemical engineers who learned physical chemistry at the same time as organic chemistry, such ideas as free energy, entropy, and probability distribution are quite familiar. But even premedical students know how to drive and know that their car will roll downhill on an incline if they shift into neutral and release the brake. It is easy to generalize from this commonplace experience to the statement that all

[1] Presented before the Division of Chemical Education at the 119th Meeting of the American Chemical Society, Boston, April, 1951.

physical systems if left to themselves will seek out the lowest energy level accessible to them. We drive this home thoroughly before resonance is ever mentioned. Then, having made clear on a number of examples that resonance means that the properties of the actual molecule are intermediate between the properties we would read off from any of the component structures, we raise the question of the energy content of the resonance hybrid. Using the diagram shown in the figure, in which energy levels are represented on the ordinate axis and E_1 and E_2 are the energy levels of two contributing structures I and II (for the sake of simplicity we discuss only the case of a hybrid of two structures), we ask the



students: Where in the diagram should the energy level E of the hybrid be represented? The first reaction of most students, after having been indoctrinated on the properties of the hybrid as intermediate between those of the component structures, is, of course, to say that E is intermediate between E_1 and E_2 (dotted line); but it is easy to show that this is a fallacy by calling on the second of our fundamental principles, which would require the molecule to change from the hybrid state to state I if the energy level of the latter (E_1) were lower than that of the hybrid (E). In this case obviously structure I would represent the molecule exclusively and there would be no resonance. If there is resonance —as we are led to postulate from experimental evidence—it is also a necessary logical conclusion that E, the energy content of the hybrid, must be lower than E_1; resonance energy, and the greater stability of the hybrid, follows as an inevitable corollary of the concept of resonance.

Next we proceed to an inquiry into the importance of the relative contributions of the contributing structures. To this end, we imagine that E_2 is moved up on the y axis to ever higher energy levels. Always relying on our second fundamental principle, we conclude that concurrently structure II becomes more and more improbable and its contribution to the state of the molecule less and less important. In the extreme case the contribution of structure II is zero, the molecule is completely represented by structure I, and we are dealing with a nonresonating molecule. Without resonance, there is, of course, also no resonance energy. We now reverse what we did before and imagine E_2 coming down on the y axis; as E_2 comes closer to E_1, the contribution of structure II to the state of the system, *i. e.*, its resonance with all its corollaries (its resonance energy, in particular) increases. The maximum resonance and the maximum resonance energy are attained when E_1 and E_2 exactly coincide: resonance and resonance energy are at a maximum if the energy levels of the contributing structures are identical, a case which certainly arises when the two structures are identical.

We now suppose that there is a third contributing structure. We repeat our earlier reasoning, substituting the hybrid for structure I and the third contributing structure for structure II. We arrive by the same logical steps at the conclusion that the contribution of a third structure necessarily requires an energy level even lower than E, and likewise for each contributing structure: the more structures contributing to the state of the molecule the greater its resonance energy.

Thus we are able to achieve the gratifying result that we can derive the concept of resonance, as well as all the most important statements about its bearing on the energy of the hybrid, and the influences which make for maximum resonance energy, by plain reasoning from experimental facts and from fundamental laws of physics and physical chemistry. Nobody claims, of course, that such reasoning can be a substitute for quantum mechanics and the insight it affords. But if we are faced with the choice of either forcing our students to learn quantum mechanics when they are not ready for it or making them accept dogmatic statements handed down from esoteric regions which must forever remain closed to them, it is pleasant to be able to find a third alternative which gives the student the satisfaction of arriving at all the elementary essentials by his own reasoning powers.

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TRACER ISOTOPES IN BIOCHEMISTRY

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IN SCIENCE, as in other fields of endeavor, the faith of persons in its future is quite varied. A well-known Frenchman once made statements to the effect that there are certain topics of a scientific nature concerning which the human race will forever be constrained to ignorance. Unfortunately he saw fit to illustrate his remarks by taking as an example the topic of the chemical constitution of the stars. Not long afterward, the new science of spectroscopy was introduced.

The men who developed radar probably conceived of it in its similarity to radio or as a navigational device. I doubt that they ever thought of it at first as an instrument which might be used for chemical analysis.

Similarly, back in the early 1900's when J. J. Thomson was performing his experiments with the positive rays which enabled him to determine atomic masses, I am sure he was not thinking of the intermediary metabolism of the sterols or of the length of life of the hemoglobin cell in the blood stream. Yet when Thomson tested ordinary neon with his positive ray apparatus and found results which could be explained only by the assumption of the existence of two neons with distinct masses, he was laying the cornerstone for a powerful technique in the attack on problems in biological chemistry.

Thomson was an important man in his time. So much so that a well-known American physicist speaking before the American Physical Society about 15 years ago and referring to the prerelativity, prequantum theory, "common sense" era of physics, called it the "Vintage of J. J. Thomson."

We seem to have come a long way since the "Vintage of J. J. Thomson." The word, "isotope," is now a household word among scientists of all kinds. The positive ray apparatus of Thomson, modified by other experimenters such as Aston and Dempster into the modern mass spectrographs, has been employed repeatedly to investigate the chemical elements. More than 250 naturally occurring isotopes have already been identified and over twice this number have been induced by bombarding atomic nuclei with rapidly moving particles (*1*).

ISOLATION AND DETECTION

In 1919 Rutherford produced oxygen of mass 17 by bombarding ordinary nitrogen with alpha particles. This is a stable isotope. Radioactive isotopes are frequently produced by particle bombardment, as in the production of P^{30} by bombarding aluminum with an alpha particle. The phosphorus disintegrates into Si^{30} by emitting a positron.

The particles used for bombardment include protons, deuterons, neutrons, and alpha particles. Electrons are too light in mass to do the requisite damage. The charged particles can be accelerated by devices such as the linear accelerator or the cyclotron, thus making the bombardment more effective. Neutrons, being without charge, cannot be accelerated by electric or magnetic fields and must be used when produced in order to get them at high speeds. If slower neutrons are desired, material to check their speed can readily be introduced. Neutrons can be produced, for example, by allowing alpha particles to strike nuclei of beryllium. It so happens that the atomic pile reactions involve neutron bombardments of uranium. Such a pile, while limited to neutron bombardments, is nevertheless a more intense source of isotopes than the cyclotron (*1*). Some isotopes are not obtained in elemental form but occur within compounds. Carbon 11 which is used in biochemical research is obtained from boron or boron oxide by bombardment with deuterons (*2*). The carbon is produced in the form of boron carbide or of the oxides of carbon.

On the whole it can be said that modern isotope production and separation is a result of the discoveries of the neutron, heavy hydrogen, and artificial radioactivity in the 1930's and the development of the cyclotrons and atomic piles which followed (*3*).

Isotopes, to be useful in biochemical research, must be later detected in body materials or excreta. The radioactive isotopes are easier to detect in minute quantities than are the stable isotopes, but have the disadvantage in that too much radioactive material may be harmful to the organism. In addition the half-life of radioactive substances imposes time limits which make the use of certain isotopes prohibitive. Stable isotope detection is reasonably sensitive, however. Amino acid samples with a 50 per cent increase of the stable isotope N^{15} can be diluted 20,000 times before it escapes detection (*4*).

Carbon 11 has a half-life of 20 minutes (*2*), yet this isotope is often used biochemically due to the fact that tests for radioactive isotopes are generally sensitive enough to follow them for 5 to 7 times the duration of the half-life (*4*). A decay curve should be plotted for a separate sample of the isotopically labeled material while the experiment is going on. Where more than one radioactive isotope is present in the experimental material a mixed decay curve is needed, which will automatically be produced by the control material from which the curve is obtained (*5*).

Many isotopes have more convenient half-lives than

does C^{11}. There is an iron isotope with a half-life of 47 days and one of sulfur with 80 days (*5*).

Apparently the organism cannot distinguish between these isotopes. Ordinary tap water contains deuterium in a 1 to 5000 ratio with protium (the hydrogen of mass 1) and since water from animal organisms has been found to have the same density as tap water, there is no evidence of isotope discrimination on the part of the animal (*6*).

Since organisms make little or no distinction between isotopes, it is evident that isotopically labeled materials must be isolated by nonbiological means. Fortunately there are several properties, largely physical in nature, with respect to which isotopes differ. The mass spectrograph itself has been adapted to isotope separation. Of course the very process of isotope detection which was used by Thomson and his followers involves the separation of isotopes, but in such minute quantities as to be of value only in making photographic records. Since 1941 this so-called electromagnetic method of separation has been improved so that actual samples of the isolated isotopes may be collected (*7*). The yield is still not high, however.

The majority of the methods used for isotope separation are statistical in action. They depend upon properties in which the isotopes differ so slightly that a clear-cut separation is not possible; but a variation in isotope composition between two samples may be obtained and by a repetitious procedure almost any degree of purity may be attained. Statistical methods have the disadvantage that the number of repetitions needed for any useful degree of purity is large. Included in this category are the diffusion methods, distillation, electrolysis, and methods of chemical exchange.

The gaseous diffusion method was demonstrated in 1896 by Lord Rayleigh and was used by Aston in the first partial separation of isotopes (neon) (*7*). Complete neon separation was later obtained by others using this method, but recycling the separated gases back through the diffusion membranes (*7*). This method depends on Graham's law which states that the speeds of diffusion of gases are inversely proportional to the square roots of the molecular weights.

The thermal diffusion method was introduced for this purpose in 1938 by Clusius and Dickel in Germany (*7*). They used a vertical tube with a wire along the axis which was kept 600°C. hotter than the tube walls. Material may be fed to such an apparatus in either the gaseous or liquid state (*8*).

Tall cylindrical centrifuges may be used to produce isotope separation (*7*). Heavy molecules will increase in the region around the periphery and light molecules near the axis. Fractional distillation can be employed for the separation of light and heavy water as the boiling points differ by 1.4°C. (*7*). The two water isotopes can also be separated by electrolysis. An increased concentration of heavy water has been found in cells used for the commercial production of hydrogen and oxygen by electrolysis (*7*).

While the gaseous diffusion and electromagnetic methods were particularly used for the separation of uranium isotopes in connection with the atomic bomb they are limited in usefulness when it comes to isotopes of biochemical value (*8*). A very satisfactory method for these is that of chemical separation. Hydrogen, carbon, nitrogen, oxygen, and sulfur compounds have been produced in this manner (*8*). A chemical exchange method is involved. In simple exchange reactions between compounds of two different isotopes, the equilibrium constant is not exactly one (*7*). In the catalytic exchange of hydrogen atoms between hydrogen gas and water, the water will contain three or four times the heavy hydrogen concentration that will be found in the hydrogen gas in equilibrium with it (*7*).

In the detection of materials labeled with isotopes, just as in their isolation, the biochemists need the assistance of the physicists. All isotopes can be separated by means of the mass spectrometer. Hence the proportion of the different isotopes can be detected by this means whether the isotopes are radioactive or stable. In the case of the radioactive isotopes, however, the detection is easier and more sensitive when methods utilizing the emitted radioactivity are employed. Such methods involve the use of the electroscope, electrometer, or Geiger-Müller counter. Radioactive isotopes may emit alpha, beta, or gamma rays and in some cases positrons. But most artificially produced radioactive isotopes used in medical experiments happen to emit beta rays (*9*). Some emit gamma rays in addition. Biochemists are consequently interested in beta and gamma ray detection.

As beta rays which are electron streams pass through air or other gases they ionize the atoms or molecules with which they collide. Each particle ionized splits into one positive and one negative ion. Under normal temperature and pressure conditions, 50 to 150 ion pairs are formed as the beta particle or electron traverses each centimeter of its path (*9*). The length of the beta ray path is nearly independent of the material through which it passes (*9*). Beta rays are detected electrically by the magnified charge resulting from all the ion pairs produced. The ray is allowed to pass through the gas in an ionization chamber which is attached to an electroscope or an electrometer in such a manner that the electroscope will deflect in proportion to the charge present in the chamber. Some electroscopes are sensitive to 10 beta rays per second through the ionization chamber (*9*). While the simplest electroscopes operate at atmospheric pressures, it is possible to increase the sensitivity by having the ionization chamber kept at an elevated pressure. More ion pairs will thus be produced. With argon in the chamber at 60 pounds per square inch above atmospheric pressure, an electrometer can be increased in sensitivity by a factor of 5 to 10 (*9*).

A greater increase in sensitivity results from the use of a cylindrical ionization chamber which has negatively charged walls containing a positively charged axial wire. This apparatus is sealed and the voltage between electrodes kept at one or two thousand volts

according to size and spacing and to pressure and nature of enclosed gas (*9*). This is the Geiger-Müller counter. A single electron will cause a discharge between electrodes. These discharge pulses are weak but are amplified by electronic tube amplifiers and the number of pulses are either counted directly by an electrical counter or counted as multiple units by the use of a scaling circuit (*9*). A counter of this type was first tried in 1908 by Rutherford and Geiger (*10*). Korff tells of one laboratory which exhibits a fork and a spoon suspended in the air and having a potential difference between them. This emphasizes the bare essentials of a Geiger counter, so to speak. One merely needs two electrodes at different potentials with some gas between them.

The increased sensitivity of the Geiger-Müller counter over the electroscope or electrometer is that the secondary electrons, or those produced by the beta rays as they ionize the gas, will themselves act to produce further ionization (*10*).

If the radioactive material to be detected is itself a gas, it is introduced directly into the counter in the presence of argon and alcohol (*9*). If a liquid is to be examined the counter with a thin-walled glass jacket is immersed in it. And if the material to be tested is in solid form it is placed directly in contact with a thin mica window at one end of the counter. Counters for liquids or solids may contain hydrogen or helium. The helium is common because it can be kept at atmospheric pressure while using an electrode potential difference which is lower than with other suitable gases.

The counter should be standardized periodically and the background radiation (mostly cosmic rays) should be determined and corrected for (*5*).

Gamma rays can be detected as are beta rays because of their production of ionization and hence of secondary electrons (*9*). Gamma rays being electromagnetic waves will, of course, not be detected directly by this type of apparatus. Should it be necessary to use a radioactive substance which emitted neutrons, these could be counted by a Geiger-Müller counter filled with boron trifluoride gas (*11*). Neutrons striking the boron will result in the emission of alpha particles which can then be detected by ionization.

Ionization will not be produced by stable isotopes and one must resort to mass methods. The mass spectrometer is commonly used for stable isotope detection. Theoretically the isotopic composition of an element could be determined by atomic weight determinations of the mixture. This requires much time and labor and samples of large size, which even then are not sensitive to small isotopic variations (*12*). A sample of 0.5 ml. of nitrogen is, on the other hand, sufficient for mass spectrometer analysis (*11*) and the spectrometer is sensitive to 1 per cent (*12*). The sensitivity is effectively decreased to 3 per cent by impurities present in the reagents used to treat the material (*12*).

N^{15} is frequently used as a label. Organic compounds containing this nitrogen are changed to nitrogen gas by first getting the nitrogen into ammonia by the Kjeldahl process and transforming this into nitrogen by the following process (*12*):

$$2NH_4^+ + 3BrO^- \rightarrow N_2 + 3Br^- + 2H^+ + 3H_2O$$

The nitrogen is then introduced into a vacuum tube containing the mass spectrometer.

Certain organic substances are sometimes determined quantitatively by adding an isotopically labeled sample of the compound to the material being studied. A sample of the desired substance is then isolated by chemical means. The increase in proportion of labeled isotope in the isolated sample should not differ from its proportion in the unisolated part of the material since ordinary chemical methods do not distinguish between isotopes. The amount of the original substance in the original mixture (y) can then be determined (*13*) from the equation:

$$y = \left(\frac{C_0}{C} - 1\right)x$$

where x is the amount of labeled material added, C_0 is the isotope content above normal of x, and C is the isotope content above normal of the isolated sample. A sample of from 5 to 100 mg. is needed for this analysis.

A new technique discovered during the war may be useful in making biological studies with stable isotopes. It was found that atmospheric gases absorb certain radar wave lengths and that microwave transmitters and receivers can be designed to form a microwave spectroscope for the detection of the absorption spectra of these gases (*14*). This apparatus can now be used with samples of skin, hair, or nails to detect isotopes from their microwave absorption spectra.

A method suitable for the detection of deuterium or heavy hydrogen depends upon a difference in refractive index between ordinary and heavy water (*15*). This difference is 0.00462 for the D line of sodium at 20°C. This method is accurate to 0.02 per cent in the Zeiss interferometer providing the percentage of deuterium in the sample multiplied by its weight in grams exceeds 0.2.

An even more sensitive method depends on the difference in density between ordinary water and heavy water (density 1.1079) (*15*). A density change can be determined to 0.5 part per million which corresponds to 0.0005 per cent deuterium, by observing the position of a float through a microscope.

ISOTOPES IN METABOLISM STUDIES

In metabolism studies, the aim is to follow through the body various substances which were labeled before ingestion. The use of rare isotopes for labels has proved to be the ideal technique since, in general, the body does not discriminate between isotopes. Labeling by using compounds differing from the usual ones by the addition of extra methyl or halogen groups has been tried but found unsuccessful as the body treats such derivatives in a different manner from their related compounds (*6*).

Isotopes useful in metabolism studies include the rare stable isotopes, H^2, C^{13}, N^{15}, and O^{18}; the long-lived

radioactive isotopes, H^3, C^{14}, P^{32}, and S^{35} (*16*); and to some extent the short-lived radioactive C^{11} (*17*). Where both radioactive and stable isotopes of the same element are available, each can be used separately as a check on the other. This can be done, for example, with C^{13} and C^{14}.

Radioactive isotopes are limited in use by the presence of nuclear isomerism (*16*). An unstable nucleus may exist in two or more isomeric forms with different radioactive properties. Br^{80} has one half-life of 4.4 hours and another of 18 minutes. The former changes to the latter which then changes to krypton. Co^{60} has a half-life of 10.7 minutes and another of 5.3 years. Most of the former decays to form the latter but some of the 10.7-minute variety will decay directly to nickel as does all of the 5.3-year variety. When one nuclear isomer goes to the other form it may be ejected completely from the chemical compound because of recoil from the energy of a departing gamma ray. This energy may be great enough to break the chemical bond.

Synthetic optically active compounds are racemic mixtures. Since the optical isomers differ in solubility, a separation can be effected and such a separation may be desirable as it permits the use of only the natural isomer (*13*).

Inorganic tracers are useful, although sometimes less interesting than those of an organic nature. Microscopic amounts of boron, manganese, and copper can be studied and detected by using labeled samples (*3*). The absorption of iron in mammals has been studied and it is known that iron which enters the mouth in the ferric form is reduced to ferrous in the jejunum and absorbed in the intestinal mucosa where it combines with protein to form ferritin. It is stored as such but is in equilibrium with an oxidized form of iron (ferric) in the blood. Radioactive phosphorus has also been used in the investigation of inorganic metabolic processes (*18*).

The organic part of a compound is generally labeled either as to nitrogen or carbon. Deuterium which remains attached to a carbon atom may be used as a carbon label. Occasionally both the carbon and the nitrogen in the same molecule may be labeled, as in the multiple labeling technique of Schoenheimer and his collaborators (*16*). S^{35} has been used as a label for sulfur containing amino acids and proteins (*17*). O^{18} in OH groups of organic compounds is stable and can be used as a label, but in ketone groups it is unsatisfactory as it exchanges with the oxygen in any water which may be present (*19*).

Among the carbons, C^{14}, which is a radioactive isotope of long half-life, is becoming most important. Unfortunately the rays emitted are too soft to detect with a Geiger-Müller counter held externally to the body as with C^{11}, but there is no time limit (*17*).

In using deuterium as a carbon label, one is somewhat limited by the differences in carbon-hydrogen bond strength with different hydrogen isotopes. The deuterium-carbon bond is stronger than that of carbon with ordinary hydrogen (*16*). Carbon-deuterium bonds in amino acids have been found stable enough for metabolic studies (*20*) despite a slow exchange with hydrogen of water which was found in some cases (*21*). The carbon-hydrogen bonds in fatty acids and sterols are also generally very stable (*20*) although these substances, when isolated from animals which had drunk heavy water, were found to contain some deuterium (*21*). Nevertheless the fatty acids and sterols will not exchange carbon-bound hydrogen with water even when heated for prolonged periods in the presence of acid or alkali. Labile hydrogen as in the COOH, OH, NH_2, and CHO groups will exchange with water and therefore such groups should not be labeled with deuterium as a carbon label (*6*).

Gaseous nitrogen has been found not to exchange with that in ammonia and the nitrogen of one amino acid will not exchange with another amino acid in aqueous solution at 100° (*19*).

ISOTOPIC COMPOUND FORMATION AND RECOVERY

In the recovery of isotopic compounds, care in avoiding contamination is not necessary (*5*). Contaminants will contain the ordinary isotopic ratios. The recovery process may be difficult when the labeled material is widely dispersed in the tissues. In such cases the separation may be assisted by adding a quantity of non-labeled material of the same chemical nature.

It may be of interest to examine the preparation methods for a few of the labeled organic compounds used in biochemistry. Lactic acid labeled with the short-lived radioactive isotope C^{11} has been prepared using the labeled carbon both in the carbonyl group and in the alpha or beta position. The former is prepared by the reactions (*2*):

$$C^{11}O_2 + K + NH_3 \rightarrow KC^{11}N$$
$$KC^{11}N + CH_3CHO \rightarrow CH_3CHOHC^{11}N$$
$$CH_3CHOHC^{11}N + H_2O \rightarrow CH_3CHOHC^{11}OOH$$

and the latter as follows (*2*):

$$Na_2CO_3 + BaCl_2 \rightarrow BaCO_3 + 2NaCl$$
$$Na_2C^{11}O_3 + BaCl_2 \rightarrow BaC^{11}O_3 + 2NaCl$$
$$BaC^{11}O_3 + BaCO_3 + Mg \rightarrow BaCC^{11} + MgO$$
$$BaCC^{11} + H_2O \rightarrow HC{\equiv}C^{11}H$$
$$HC{\equiv}C^{11}H + H_2O \rightarrow CH_3C^{11}HO$$
$$HC{\equiv}C^{11}H + H_2O \rightarrow C^{11}H_3CHO$$
$$CH_3C^{11}HO + KCN \rightarrow CH_3C^{11}HOHCN$$
$$C^{11}H_3CHO + KCN \rightarrow C^{11}H_3CHOHCN$$
$$CH_3C^{11}HOHCN + H_2O \rightarrow CH_3C^{11}HOHCOOH$$
$$C^{11}H_3CHOHCN + H_2O \rightarrow C^{11}H_3CHOHCOOH$$

Radioactive samples of alpha ketoglutaric acid are obtained from incubation of liver tissue with pyruvic acid in the presence of radioactive carbon dioxide (*22*):

$$\begin{array}{ccc} {}^{*}CO_2 & & {}^{*}COOH \\ + & & | \\ CH_3 & \rightarrow & CH_2 \\ | & & | \\ CO & & CO \\ | & & | \\ COOH & & COOH \end{array}$$

Deuterium gas is produced by electrolysis of a dilute solution of deuterium sulfate (sulfuric acid with heavy

hydrogen) in heavy water (*15*). The substances to be hydrogenated with this deuterium must be in the form of esters or other derivatives in which there is no labile hydrogen.

Amino acids containing isotopic nitrogen may be obtained by the catalytic hydrogenation of alpha keto acids in the presence of labeled ammonia or by the coupling of alpha bromo esters with labeled potassium phthalimide (*23*).

RESULTS OF ISOTOPIC STUDIES

A complete statement of the results would be too extensive for this paper. Some typical studies involving fats, carbohydrates, amino acids, and sterols are included.

Fat studies have been accomplished by labeling the fat carbons with heavy hydrogen. The so-called deuterated fats were prepared by catalytic hydrogenation of such unsaturated fats as linseed oil (*22*). The hydrogen used contained a high concentration of the heavy isotope. Mice were fed the labeled fats for several days before being killed. Fat deposits in the body were later burned to determine the percentage of heavy hydrogen in the water formed. Most of the deuterium was found in fat deposits and very little in other tissues. A small amount was found in the form of heavy water in body fluids (*24*).

Interesting carbohydrate experiments have been carried out with C^{11}. About two hours were consumed in preparing a sodium lactate labeled with this isotope (*25*). The material was fed to rats for 2.5 hours before they were killed. The air expired from the lungs was examined for evidences of radioactivity during life. Such radioactivity was detected very soon and increased during the second and third half hours of feeding, although the period of greatest radioactivity differed from animal to animal. It was still present when the animals were killed. The liver glycogen was then isolated and tested and the results found to support a hypothesis of the mechanism of glycogen formation (*26*). It had been predicted according to this hypothesis that glycogen from rats fed with carboxyl labeled lactate should contain half as much C^{11} as that from rats fed with alpha or beta labeled lactate.

Studies involving labeled carbon dioxide are obtained by injecting rats with radioactive bicarbonate. C^{11} has been used here as a label. When fasted rats were fed lactic acid by stomach tube and injected with the radioactive bicarbonate, there was evidence of the appearance of C^{11} in glycogen of the liver, but not in muscle glycogen (*27*). The scientists who conducted these experiments concluded that muscle and liver glycogen were of a different origin and did not exchange between themselves. On the other hand when the radioactive carbonate was injected into fasted rats which were being fed glucose, there was a little C^{11} in the muscle glycogen as well as a larger portion of it in the liver (*28*). The results of the experiments on the formation of liver glycogen have led to the hypothesis that the different chemical substances which cause an increase in liver glycogen may be considered interchangeable if they are capable of being brought into enzymatic equilibrium in the body with pyruvic acid. It is the pyruvic acid which condenses with carbon dioxide to start the formation of glycogen (*27*).

$$C^{11}O_2 + CH_3\overset{\overset{O}{\|}}{C}COOH \rightarrow HO\overset{\overset{O}{\|}}{C}{}^{11}CH_2\overset{\overset{O}{\|}}{C}COOH$$

Oxalacetic acid

A number of important phenomena characteristic of amino acids and proteins have been discovered or substantiated by isotope experiments. It is evident that amino acids may replace each other in body proteins. This may occur because of a complete breakdown and resynthesis of the protein, or a replacement of amino acids involving the opening and closing of peptide linkages, or because of chemical changes taking place at the nitrogen-containing groups (*29*). It has not been possible to ascertain which type of reaction is taking place. The stable isotope N^{15} is generally used as a nitrogen label. In a 10-day experiment involving the feeding of tyrosine labeled in this way, 25 per cent of the N^{15} was found in the proteins as tyrosine and 75 per cent had evidently been transferred to other amino acids (*30*). In a three-day experiment with labeled leucine, it was found that there was a considerable replacement of leucine in proteins by new leucine molecules (*29*). Thirty-two per cent of the labeled leucine was found in the proteins. More must have been introduced into the proteins as some of it was no doubt itself replaced. Some of the alpha amino nitrogen was transferred to other amino acids. By labeling the leucine with deuterium as well as N^{15} it could be determined that some of labeled leucine had accepted nonlabeled nitrogen from other amino acids. Continuous chemical processes are occurring to a considerable extent in tissue proteins even under conditions of nitrogen balance.

Rats are able to utilize dietary ammonia in the form of ammonium citrate for amino acid and creatine synthesis. Some of the labeled nitrogen from such experiments has been found in arginine but entirely in the guanidine group (*22*). When the arginine was separated into ornithine and ammonia with strong alkaline hydrolysis, all labeled nitrogen was in the ammonia. When creatine with N^{15} in the amidine group was administered, the creatinine formed from it was isolated from urine. It contained the N^{15} in the same position in the amidine residue. Creatine from the muscles was found to contain N^{15} in the amidine group when arginine with N^{15} in the amidine group had been fed. When N^{15} glycine was used, the muscles also contained N^{15} but this time it was not in the amidine group. Evidently the amidine group is stable.

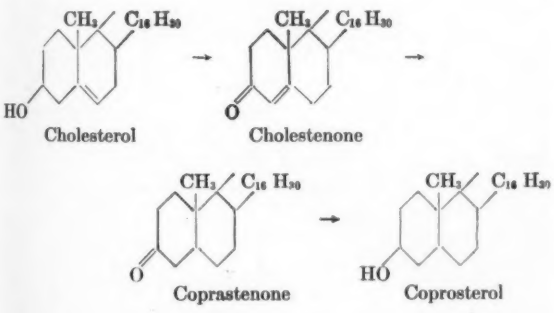
When creatine formation involves methionine labeled with heavy hydrogen in the methyl group, it is still found in the methyl group of the creatine (*22*). Methionine labeled with radioactive sulfur has been given to animals. It can later be found in cysteine and cystine which are isolated from the tissues.

In addition to creatine and arginine, glutamic acid,

aspartic acid, histidine, and lysine have been isolated from animals fed with ammonium citrate (*22*). Glutamic and aspartic acids displayed evidence of high metabolic activity by the high isotope concentration which they contained (*31*). All other amino acids studied had significant N^{15} concentration with the exception of lysine (*22*). Lysine is an exception probably because it does not undergo deamination. In another experiment deuterium was introduced into the body fluids of mice and amino acids later isolated (*21*). All acids isolated contained carbon-bound deuterium with again the exception of lysine. It is interesting that the amount of deuterium in these isolated acids was higher than found *in vitro* by an exchange with heavy water at elevated temperatures. The deuterium in the acids from the rats was stable and probably not due to an exchange reaction with heavy water but to a deamination and reamination, or to enzymatic reactions in which the carbon-bound hydrogen is labilized.

Labeled benzoic acid given to mammals was detoxified by being coupled with glycine in the formation of hippuric acid (*32*). This reaction took place in the absence of dietary glycine but the tolerance for benzoic acid was increased when dietary glycine was present. The amount of glycine furnished by the animal was greater than the amount in the body and was apparently synthesized by the animal.

It will be fitting to conclude this paper with a group of studies carried out to test a theory advanced by Schoenheimer, Rittenberg, and Graff according to which the biological conversion of cholesterol to coprosterol is not a direct hydrogenation of the double bond, but involves first an oxidation to cholestenone followed by a reduction to coprastenone and a further reduction to coprosterol (*33*). This is in agreement with the laboratory process.



Cholesterol Cholestenone

Coprastenone Coprosterol

The feeding of unlabeled coprastenone and cholestenone may produce an increase in the excretion of coprosterol without being involved in the reaction (*33*). Both deuterium-labeled cholestenone (*34*) and coprastenone (*33*) have been fed to dogs in separate experiments. In each case the coprosterol excreted contained deuterium. Cholesterol can be separated from coprosterol by brominating the former, which is unsaturated (*35*). In the brominated condition it will not be precipitated by digitonin. The coprosterol, being saturated, will not be attacked by the bromine and will be precipitated by the digitonin.

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LOUIS HACKSPILL

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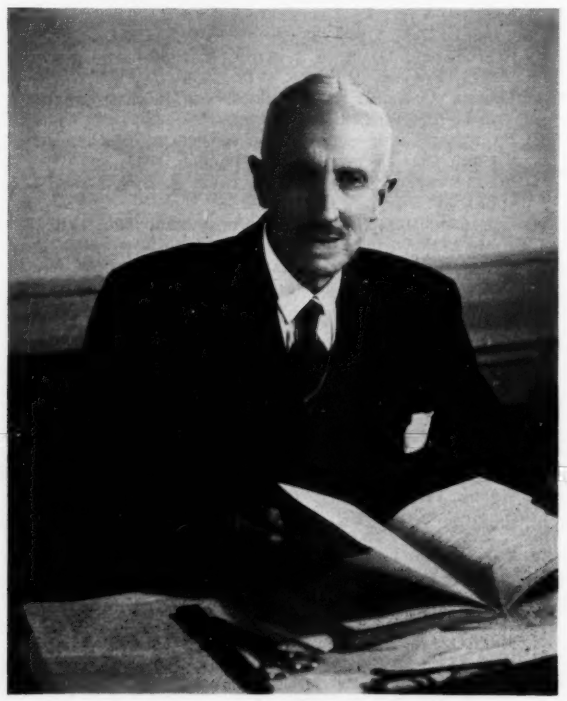
ALTHOUGH his grandfather and father were professional soldiers, Louis Hackspill did not inherit a taste for arms. His teacher in the secondary school had awakened an interest in chemistry, and his understanding father permitted him to set up a laboratory in the summerhouse at the foot of their garden in Versailles. Here young Hackspill and several of his classmates met on Thursdays and Sundays to repeat the demonstrations which M. Fontaine used to illustrate his lectures on chemistry, and to perform other experiments including, of course, some involving explosive mixtures.

After Hackspill (born at Paris, May 3, 1880) finished the lycée in 1898 he took courses at the University of Paris and also enrolled in the "Enseignement pratique de la chimie appliquée," which had been organized three years previously by Friedel, and which afterwards became the Institut de Chimie of the University of Paris. He was awarded the diploma of ingénieur chimiste in 1903 by the Institut de Chimie appliquée,



Louis Hackspill

then headed by Moissan who, appreciating his promise, gave him the post of private *préparateur* in his own laboratory. Moissan, then at the height of his glory, was submerged in administrative duties; he was rarely seen in the laboratory, which was essentially headed by P. Lebeau. Here Hackspill learned how research is really conducted. After Moissan's death in 1907, Hackspill accepted the post of *préparator* to Pechard, with whom he worked at the University of Paris until 1913. When he came to Pechard, the latter said: "My laboratory man has had long practical experience in preparing demonstration experiments. Let him do it, he can get them ready far better than you can. Accordingly, you may devote the major part of your time to research. That is all I ask of you, but it will be necessary for you to work and produce." The doctorate was awarded by the University of Paris in in 1907, and the Docteur ès Sciences in 1912.

In 1913, Dr. Hackspill was named to the chair of chemistry at the University of Nancy. Ten months later World War I began. He was mobilized as lieutenant on August 2, 1914; he took part in the battles of the Meuse and the Marne. The Croix de Guerre was awarded for bravery in 1916. Only on direct order did he (promoted to a captain in the field) leave the front to place his chemical abilities at the service of La Patrie. He perfected the charging of gas containers; proved the possibility of transporting bromine in iron bottles; developed the drying of chlorine by iron, the ferric chloride acting as desiccant; and worked out a method, later adopted by industry, for the preparation of stannic chloride. After the Armistice (November 11, 1918), he was appointed inspector of the synthetic ammonia plant at Oppau. This experience led to his book "L'Azote," published in 1922. In 1919 he was put in charge of the publication of the reports on the German chemical industries. The Cross of the Legion of Honor, awarded in 1920, was the public recognition of these many valuable services. His "La petite industrie chimique" (with Rémy-Genneté) appeared in 1926.

The return of Alsace to France brought with it the complete reorganization of the University of Strasbourg, which was reopened in November 1919. Hackspill, a scion of an old Alsatian family, was delighted to accept the chair of inorganic chemistry, and to give the course in applied chemistry. His lectures in polished but simple language were illustrated by demonstrations on the grand scale, such as precipi-

tations carried out in 3-liter flasks. Though sympathetic and friendly with his students, he quickly showed his superiority by insisting that they complete in creditable fashion several assignments in glass blowing, an art in which he was so competent that he made even difficult constructions seem easy. Because he never lost sight of the practical side of chemistry, he soon gained the confidence of the industrialists of the region, many of whom donated funds for the support of his researches. They also opened their plants to his students, who thus learned of the difficulty attending the passage of a reaction from laboratory to plant scale. From 1929 to 1932 he was director of the Institut de Chimie at Strasbourg. His success as a teacher and the excellent research issuing from his laboratory made it inevitable that he would be called to a post in Paris. In 1932, at the insistence of Pechard, Hackspill became a successful candidate for his old master's post at the Sorbonne. After Urbain's death (1938) Hackspill was named (1939) to the chair of mineral chemistry and also director of the Institut de Chimie. In a sense therefore, he became the successor of his early teacher, Moissan. He also served as lecturer at the two Écoles normales supérieures. His well-earned retirement came in 1951.

During World War II, he again was in active military service. In 1939, with the rank of lieutenant colonel, he was town Officer at Hagenau in Alsace, and evacuated this citadel only after receiving express orders from above. His was the last company to leave. Before going, he organized a voluntary militia, which prevented the looting of Hagenau. During the occupation he carried on his researches at the Sorbonne.

Many honors have come to this eminent chemist. The Académie des Sciences awarded him the Cahours Prize in 1911, the Houzeau Prize in 1926, the Lacaze Prize in 1932. He was made *Officier* of the Legion of Honor when the Institut de Chimie celebrated its 50th anniversary. Election to the Académie in the section of *académiciens libres* came in 1944. In 1946 he held two important offices: President of the Societé chimique de France and President of the Féderation nationale des Associations de Chimie en France. He has been a member of the American Chemical Society since 1918.

Professor Hackspill's scientific achievements have been almost entirely in the inorganic field. He has specialized in the study of the alkali metals and their salts, the alkaline earth metals, boron, metal hydrides, deuterides, phosphides, etc. He has constructed a type of thermobalance which operates in a vacuum, and hence, by virtue of the pressure produced, can be used to follow thermal decompositions which are accompanied by the liberation of a gas.

In 1903 Moissan and Lebeau entrusted to Hackspill a specimen of metallic calcium, which they had obtained from a German works at Bitterfeld. His problem was to prepare alloys of this metal, which was quite rare at that time. Hackspill found that when it was heated *in vacuo* with the respective alkali chlorides, potassium, rubidium, and cesium were set free. By this procedure, he prepared several kilograms of the latter two metals. This reaction has been adapted to industrial purposes for preparing photoemissive cells. It has been generalized to calcium carbide, to nickel, to iron (preparation of lithium). In Hackspill's doctorate work he measured the physical constants of the alkali metals: density, coefficient of expansion, vapor tension, electrical resistance. He then studied the platinum-cesium couple, the magnetic susceptibility, and the comparative action of water and of oxygen on the alkali metals and their hydrides and deuterides. He prepared the borates, hypophosphites, and fulminates.

With Rinck he discovered the allotropic states of calcium, and with Rémy-Genneté he showed that hydrogen can combine with calcium from −80°C. on. The direct synthesis of calcium carbide was carried out with Botolfsen, and that of calcium cyanamide with Fleck. Hackspill has proposed a method for the simultaneous determination of carbon and hydrogen, and nitrogen by a gasometric procedure employing 50 to 100 mg. of an organic substance.

He refuted the existence of any significant amount of combined argon in the blood. With his vacuum balance, he has studied the thermolysis of different varieties of alumina, chlorates, perchlorates, and bromates (with the hope of discovering the perbromates).

In recent years he and his students (de Postis, Thomas) have created a new chapter in chemistry, which presented many experimental difficulties, namely, the entirely unexpected action of cesium on organic compounds. A striking example is the discovery (with Röhmer) of ethylene cesium $C_2H_4Cs_2$. It was only to be expected that Hackspill should be entrusted with the sections on rubidium and cesium in Pascal's "Traité de chimie minérale."

Summing up, his researches have been entirely original in various fields of inorganic chemistry, and some of the most simple materials have been described with rare precision by him and his students.

In 1946 Hackspill stated: "I believe that one element of success has equally been one of my faults: stubbornness. Pasteur once wrote, not to me of course, but his remark applies equally as well to all researchers: 'Allow me to give you a word of advice, which I have always forced myself to follow, namely to persist as long as possible on one subject. By perseverance in the research, one ends by acquiring what I call the instinct for the truth.' That is why, if God gives me life, I will not quickly abandon the study of the alkali metals."

LABORATORY MEETINGS SHOULD TEACH TOO[1]

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If a laboratory manual, intended for use in general college chemistry, were to be selected at random, it is more than likely that a cookbook in general chemistry would have been chosen. In spite of the awareness on the part of educators of the fact that experimentation and interpretation of data are the foundation of scientific behavior, chemistry professors seem perfectly content to assign a manual which teaches nothing, for the most part, but blind and unreasoning obedience to printed directions. It is perfectly correct to feel that the ability to follow directions must be developed in our young students of science, but more important than that is the need for teaching them how to use a free and unprejudiced mind.

It is indeed disturbing to observe how many of our manuals in current use ask the student to mix several substances in the laboratory and to report a color, odor, or precipitate formation in the space provided so conveniently on the report page. More often than not, the student is simply repeating a reaction of which he has read in his textbook and probably has seen demonstrated in lecture. The laboratory is thus being used as a place for the repetition of demonstration experiments, and the more important necessity of providing new types of educational experience, both mental and manual, is practically ignored.

I wonder how many of us remember, when we were students, preparing our laboratory reports the night before class by copying the anticipated answers from the textbook or from a report on the same experiment written by a student who had passed the course some years before. This practice not only is a mockery of the word "experiment" and a debasement of the "scientific method" we all preach, but is also an appalling waste of valuable time by the student who hands in the experiment and the professor who grades it.

A primary function of the chemistry laboratory is to encourage impartial observation and recording of facts. Let us assume that a student, following directions, is going to bubble H_2S through a solution which he obtained from a bottle labeled "antimony trichloride." He has read, of course, that an orange colored precipitate is produced in the formation of antimony trisulfide. Let us further assume that the stockroom clerk has made an error and has put the "antimony trichloride" label on a bottle which really contains cadmium chloride. Do you suppose that on the basis of the laboratory work of the student we would ever find out about this error? I believe rather that, because of the structure and characteristics of so many laboratory courses, the student not only would tend actually to see the precipitate as orange colored, but would also make certain to describe it exactly as "orange," not "yellow" or "light orange" perhaps, but exactly in the terminology employed by the author of his textbook or by his teacher—orange!

The fact which makes all of this even more pathetic is that in chemistry we have a particularly fruitful field for teaching the elements of the scientific method of investigation by direct application of its principles. So many of us will agree that if we do nothing but awaken in the student a respect and appreciation for scientific methodology, all will not have been in vain. Despite this belief, however, most of us are constantly catering to an antithetical point of view when we meet our laboratory classes. Isn't it true that if the luckless student who carried out the experiment described above had been naive enough to have called the precipitate he observed "yellow" he would simply have been marked *wrong*?

In a first course in general chemistry the student frequently finds himself face to face with concepts and usages which he feels are abstract and intangible. Proper laboratory practice can do much to dispel these mistaken notions and can present to the student an idea of the real meaning of some of these concepts.

How many students use molecular weights in solving problems without knowing how they are arrived at? How many students use the gas constant "R" as some sort of magic number believing it to have one set of units such as liter-atmosphere per mol per degree—if they have thought of it at all? How many students speak of coulombs as if these units found use only in solving Faraday's law problems and of the electronic charge as an infinitesimal quantity of almost undeterminable magnitude? The truth of the matter, in too many cases, is that students are almost completely oblivious to the meaning of these concepts which they, nevertheless, constantly use and, in general, feel that the actual determination of molecular weight, of R, etc. must be a problem of unbelievable complexity and capable of solution only by highly trained research workers with elaborate equipment.

The student must be made to realize that these concepts have real significance, real meaning, and real magnitude. They must learn, for a better understanding of the subject matter, that quite reasonable determinations of these values can be made with relatively simple apparatus; and that complex and carefully controlled apparatus for their determination are necessary only when extreme accuracy is required. They can learn of these things by being told about it over and again, but when they have actually made such determinations for themselves, then the performance of

[1] Presented at the 120th Meeting of the American Chemical Society, New York, September, 1951.

a single experiment accomplishes more in the right direction than hours of lecture. Where, but in the laboratory, do we have the facilities and the atmosphere for producing these results?

EXPERIMENTS

It may seem that some of these comments have reflected an unduly hypercritical evaluation of a situation that can easily be improved. It is true; criticism is being enunciated. The criticism, however, is not meant so much for the situation in which we find ourselves, but rather for the fact that so little has been and is being done to remedy it. It seems that if an author writes a textbook, he is duty-bound to collect experiments from manuals that are antique in their approach and scope and, with a minimum of effort, produces a "new" laboratory manual which does nothing to make even more useful a possibly excellent and admirable textbook. The experiments given here are but a few of many which have proved their usefulness over the last five years, and which might well be included in laboratory manuals.

Principal Products. When the chemistry of the oxidation states of nitrogen is taught, it is usual to stress the point that when HNO_3 is used as an oxidizing agent any combination of its reduction products may be formed. The nature of the principal reaction will depend upon several variables, including the concentration of the acid and the particular reducing agent used. For example, we usually write:

$$Cu + \text{con. } HNO_3 \rightarrow Cu(NO_3)_2 + H_2O + NO_2 \quad (1)$$
$$Cu + \text{dil. } HNO_3 \rightarrow Cu(NO_3)_2 + H_2O + NO \quad (2)$$

Although the equations, as written, do not express the fact, it is commonly stated that in any single reaction neither NO nor NO_2 is produced to the complete exclusion of the other. The equations simply show the main product which predominates in the mixture of oxides formed as reduction products. To bring this idea out more clearly to the student, one laboratory meeting is devoted to an actual experiment involving these reactions.

The student weighs a specimen of copper and dissolves it in about 1:1, HNO_3:water. He collects the gas generated in the reaction in a eudiometer which is filled with water which had previously been boiled and then cooled. The NO_2 will, of course, dissolve and so only NO is measured as a gas. From these data, the fraction of copper which reacts according to reaction (1) compared with that used in reaction (2) is calculated. In this way, the idea of "principal product" becomes something with meaning. There is no place where the "correct" answer to this experiment can be found in texts and handbooks, and since concentrations of the acid used is varied from class to class and from term to term, copying of data is minimized and confidence in one's own work is encouraged.

Another experiment dealing with the concept of "principal product" is one which involves burning of magnesium. Normally, when this metal is burned in air, the product of reaction is understood to be magnesium oxide. Little or no attention is paid to the fact that an appreciable quantity of magnesium nitride is produced as well. In the laboratory this point is investigated by the student who burns a weighed amount of magnesium in a crucible. The ash is then dissolved in dilute acid. This converts the oxide to a soluble magnesium salt while the nitride forms the ammonium salt of the acid used. The mixture is neutralized, then made alkaline with KOH in a distilling apparatus, and the ammonia is distilled into standard acid. The amount of NH_3 formed is found by back-titration and the amount of Mg_3N_2 required to produce this NH_3 is calculated. From these data it is observed that MgO is actually not the sole product, but simply the "principal product" formed when metallic magnesium is burned in air.

The Gas Constant "R". In lectures on the gas laws, the student is frequently confounded by the constant R which we develop during discussion. This situation arises primarily, it is felt, because of limited mathematical background on the part of the student. This is not due to any special complexity involved in the treatment of gas law problems mathematically, but rather because of a difficulty in the realization of what is meant by a "constant."

It is usual for us to derive the Perfect Gas Law:

$$PV = nRT = \frac{g}{M}RT$$

and also to explain that R is the factor which relates the equations of both Boyle and Charles when simultaneous variation in both temperature (T) and pressure (P) are operating on a volume of a gas (V) having a weight g and molecular weight M. Occasionally R will be expressed as:

$$R = \frac{PV}{nT} = \frac{PVM}{gT}$$

and its units found in terms of the units used to express the variables in the equation. Frequently the value of R is presented in terms of liter-atmospheres per mol per degree. The student then memorizes this number and is completely unprepared for the shock which comes later when he finds that R has a different numerical value when millimeters of mercury are used, instead of atmospheres, to measure pressure.

In the laboratory, the student is asked to collect and measure the amount of oxygen liberated upon heating a sample of PbO_2. The weight of the oxygen collected is determined from the loss in weight of the PbO_2. Now since g, M, P, V, and T are all known, the student can calculate the value of R in the more familiar laboratory units of cc.-mm. per mol per degree. He then calculates its value in different units by changing these units before substitution in the equation. The student now has a definite laboratory contact with the idea of a mathematical constant and, by its evaluation and sufficient drill in its use, he soon becomes better equipped to realize its utility.

The Charge on the Electron. Through no fault of his own, in speaking of the charge on a single electron, the student feels that this is a value so minute and so imponderable that its actual determination is possible only by the use of elaborate equipment by highly skilled technicians. This attitude then serves also to give the quantity merely a "theoretical" aspect without any reality. Of course, precision work is required for reliable results, but in our general laboratory course we have determined the value successfully, with good results, by the use of the following very simple set-up.

A bath of cupric sulfate is electrolyzed using a polished copper cathode. This cathode is weighed before and after electrolysis. The amperage during the experiment and the time of electrolysis are carefully recorded. These data give us the *number of coulombs* required to discharge the weight of copper deposited. From Avogadro's number we obtain the *number of copper atoms* represented by that weight of copper. Since each cupric ion required two electrons for deposition, the *number of electrons* involved is then known. The charge (in coulombs) carried by one electron is readily determined. This experiment has been particularly successful in giving reproducible and acceptable values for e and provides a problem of real interest to the student.

In teaching reactions of the elements, primary consideration is given to the *use* of these reactions. For instance, in the identification of pure or mixed unknowns, the analysis of alloys, fertilizers, and baking powders, the reactions are first performed briefly and then analyses are conducted on unknowns making use of these reactions; in some cases the procedures having been developed by the students themselves. For the purpose of brevity I should like to mention only the subjects of some other laboratory experiments which we have used namely: heat of a chemical reaction, determination of water hardness, "apparent" degree of ionization of a salt, etc.

It is felt that these experiments tend to encourage a spirit of inquiry in the student and to emphasize the importance of experimental techniques. Many different analytical tools, such as gravimetric, volumetric, and colorimetric methods, are employed and in an experiment on equivalent weights the student determines the weight of a piece of magnesium not by weighing it, but by measuring the hydrogen which it releases from HCl.

It is not the contention of this paper that all of the experiments employed are original—much has been borrowed from other sources and we encourage others to borrow from us. The important thing is for more of us to realize the significance of the laboratory meetings in general chemistry and to make them the useful sessions which the time allotted to them justifies. This can be accomplished by the application of individual ingenuity to commonplace experiments and the free interchange of ideas which have proved worthy. In closing, I would like to mention a small point which should be of interest to most teachers—the grading of reports on this type of work takes less than 1 per cent of the time consumed in the grading of the old cookbook experiments.

A QUALITATIVE TEST FOR AMMONIA IN AQUEOUS SOLUTION

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THE reaction between ammonia and iodine[1] is well known, but apparently has not been utilized for detection of ammonia. We have found that this reaction affords a reliable test for ammonia which is more convenient than the usual methods of detection.[2]

One milliliter of unknown solution is neutralized to the litmus end point and two drops excess of 10 per cent sodium hydroxide solution added. Any precipitate which forms is removed by centrifuging. The clear solution is poured onto about 3 mg. of iodine on a spot plate. A crystal of iodine is pushed to the surface of the liquid with a stirring rod and allowed to slide to the bottom of the depression in the spot plate. A black, finely divided precipitate appears in the wake of the iodine crystal. With small concentrations (30 mg./liter) of ammonia, the black precipitate dissolves within a few seconds. When the concentration of ammonia is relatively high (500 mg./liter) the precipitate is more copious and persists much longer.

The test has been used successfully on a variety of solutions issued as unknowns in an elementary course in qualitative analysis and no interference by other ions has been discovered.

The black precipitate formed in the test is highly explosive when dry and should be destroyed by adding sodium hydroxide or by washing down a drain.

[1] SIDGWICK, N. V., "Chemical Elements and Their Compounds," Oxford Press, London, 1950, p. 707.

[2] FURMAN, N. H. "Scott's Standard Methods of Chemical Analysis," 5th ed., D. Van Nostrand and Co., New York, 1939, p. 630.

LABORATORY SYNTHESIS IN GENERAL FRESHMAN CHEMISTRY

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DURING the last decade, an increasing amount of attention has been given to the teaching of theoretical principles in the freshman chemistry courses. This trend has many advantages, among them a stronger basis for understanding on the part of the student. The trend has necessitated a decreased emphasis upon descriptive chemistry and upon laboratory preparations. The author feels that the pendulum should not swing so far in the direction of theory that descriptive material is practically excluded. Theory should be supplemented liberally with descriptions of interesting portions of pertinent historical information, uses and reactions of elements and compounds, and descriptions of industrial processes.

In the freshman chemistry laboratories at the University of Nebraska, the writer has found that a few experiments requiring syntheses of compounds stimulate the interest of the students remarkably and provide them with much valuable laboratory experience. Most instructors who have assigned unknowns in general chemistry or in qualitative analysis have noticed an upsurge of interest on the part of the students when their abilities are met by the challenge of the unknown. A laboratory synthesis can bring an analogous rise of interest. The student feels that he is accomplishing something and is not simply carrying out procedures, for the sake of learning, which he assumes have been established as aids to understanding the course.

The interest generated in the student is almost enough to justify this type of assignment. In addition, however, much valuable experience is received by the student. First, he is on his own. Even if the entire class is assigned the same synthesis, each student is under test to produce a pure product in good yield. Second, laboratory techniques are developed by necessity in order to get good results. A variety of operations and techniques are provided that do not necessarily arise in other types of assignment. Third, the student gets a small look at the field of laboratory synthesis, an important phase of chemical research and chemical production.

The author has used laboratory preparations for several classes involving a total of several hundred students in both of the first two semesters of beginning chemistry at the University of Nebraska. Some of the classes have been for students who have had high-school chemistry, but most of the classes have been for those who have had no chemistry training prior to university. One to two weeks (six to twelve laboratory hours) have been devoted each semester to this type of assignment.

Some laboratory manuals provide a limited number of preparations. The writer first assigned to his beginning classes such preparations as sodium hexanitrocobaltate (III) and hexamminenickel (II) bromide, given in a manual that has been used in these laboratories.[1] The methods of preparation are very similar to those in the literature.[2, 3] These two preparations include such techniques as oxidation of cobalt (II) ion to cobalt (III) ion by passage of a current of air through the solution, precipitation from 95 per cent alcohol, filtration with and without suction and both at room temperature and in an ice bath, washing the precipitated product with alcohol and ether, drying and weighing the product, and calculation of theoretical and percentage yields.

The encouraging response to this initial trial led to assignments direct from the literature. Colloidal sols have been assigned, for example, providing each student with a literature reference and requiring him to look up the reference in the library to get his procedure. An easy method of making a collodion dialysis bag and its subsequent use were explained in the lecture.[4] Each student made a dialysis bag and dialyzed his colloidal sol as part of the assignment.

Another class was assigned preparations from "Inorganic Syntheses." Each student looked up his own procedure. Potassium trioxalatoaluminate, potassium trioxalatoferrate (III), potassium trioxalatocobaltate

[1] HOPKINS, B. S., T. MOELLER, AND F. B. SCHIRMER, JR., "Laboratory and Classroom Exercises in General Chemistry," D. C. Heath and Co., New York, 1946, pp. 219–20.

[2] BILTZ, H., AND W. BILTZ, "Laboratory Methods of Inorganic Chemistry," 2nd ed., John Wiley & Sons, Inc., New York, 1928, pp. 149, 171.

[3] WATT, G. W., "Inorganic Syntheses," McGraw-Hill Book Co., Inc., New York, 1950, Vol. III, p. 194.

[4] Place a funnel in an Erlenmeyer flask of appropriate size. All equipment must be clean and dry. Pour through the funnel a small amount of collodion taking care that none dries on the walls of the flask. Up-end the flask and rotate evenly so that a uniform amount of collodion sticks to the walls of the flask and no part of the inside of the flask is skipped. Pour the excess collodion out. Blow a stream of air into the flask through a tube to dry the collodion. To remove the membrane from the flask when dry, fill the flask with distilled water, stick a glass tube down into the flask, then slit all around between the neck of the flask and the dried collodion layer to loosen the membrane. Pour the water out and pull the bag from the flask. Fill the bag with distilled water to test for leaks. It is the author's experience that the average student can make a satisfactory bag in his first or second attempt.

For dialysis, place the colloidal sol in the collodion bag and suspend in a container of distilled water. A syphon arrangement for continuous changing of the distilled water is desirable but not essential for a beginning laboratory.

(III), and potassium trioxalatochromate (III) have been assigned in this way.[5] The four preparations were divided among the members of the class. For the trioxalatoaluminate salt the student was asked to use one-tenth of the quantities specified in the procedure; for the other three salts, one-fifth of the specified quantities.

Many other syntheses from the literature are within the scope of the average freshman student. The procedures need not be difficult ones. Indeed, care should be exercised by the instructor to select preparations within the abilities of his class.

The writer requires the student to submit a report on each synthesis, giving the name of the compound, method of preparation, equations, theoretical yield, actual yield, percentage yield, discussion, and literature references. Along with the report the student submits his product properly bottled with a label giving his name, the name of the product, weight of the bottle and product, tare weight, weight of product, and percentage yield.

No claim is made as to originality in this type of assignment. The practice of giving laboratory syntheses to freshman chemistry classes, however, does not seem to be utilized to a very significant extent. Inasmuch as the author has had gratifying results in the use of a few such assignments each semester, he recommends the method as a valuable teaching device.

[5] BAILAR, J. C., JR., AND E. M. JONES, "Inorganic Syntheses," McGraw-Hill Book Co., Inc., New York, 1939, Vol. I, pp. 35–8.

A CONTINUOUS ETHER-EXTRACTION APPARATUS

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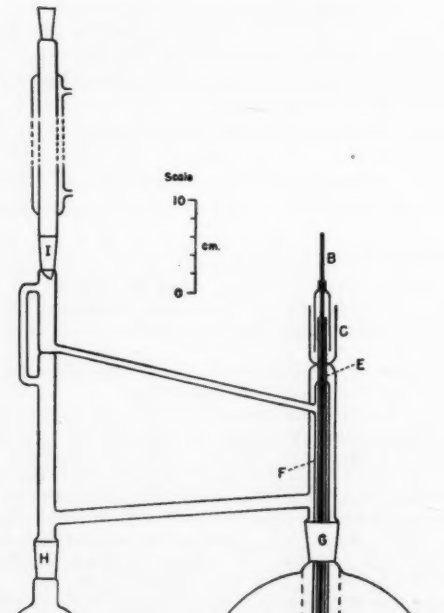


SEVERAL different designs of apparatus are available for the continuous extraction of aqueous solutions with a light solvent such as ether. Those not employing ground-glass joints suffer from the disadvantage that they require considerable time for assembly and also that rubber stoppers may be attacked by the solvent employed. On the other hand, those incorporating ground-glass joints for speed of assembly are generally designed so that a different size of apparatus must be employed for different-size extractions.

The apparatus shown in the diagram has been constructed to provide for both speed of assembly and versatility in respect of volume of solution being extracted. This versatility is achieved by keeping the height of the apparatus constant and using vessels of different diameter for the extraction chamber *A*—the figure depicts both a 5-liter and a 100-ml. vessel. Distribution of the extracting solvent throughout the solution is maintained by the stirrer *B*, through the mercury seal *C*, the solvent being fed from the reservoir *D*, by distillation, via an outer jacket *F*, to the stirrer guide *E*. *E* and *F* are sealed together at the lower end and four small holes are provided as an exit for the solvent. Separation of the two phases takes place in the upper part of *A* and the extract overflows back to the vessel *D*.

Suitable ground-glass joints are, with British Standard alternatives: *G*, 34/45 (B. 34); *H*, 24/40 (B. 24); *I*, 19/38 (B. 19).

Proceedings of the

PACIFIC SOUTHWEST ASSOCIATION OF CHEMISTRY TEACHERS

CHEMICAL EDUCATION IN SANTA BARBARA COLLEGE OF THE UNIVERSITY OF CALIFORNIA

JOHN W. SUTTON
Santa Barbara College, Santa Barbara, California

In order to meet the increasing demand for undergraduate instruction the University of California has added to its system several four-year colleges. Santa Barbara College, eighth campus of the University and the most recent of these additions, was founded in 1891 as a private school offering "instruction in cooking and manual training." It was later incorporated in the city school system, and in 1909 was established as the Santa Barbara State Normal School of Manual Arts and Home Economics.

Its curriculum was expanded to include general teacher training in 1919, at which time the name was changed to Santa Barbara State Normal School. Two years later a four-year program was introduced, and the name changed to Santa Barbara State Teachers College. With the addition of a program of liberal arts in 1935 the school became Santa Barbara State College, the designation it retained until 1944 when the affiliation with the University of California came about.

Throughout the early years of the college, chemistry functioned only in a service capacity to the major fields. Courses were presented in inorganic, organic, physiological, food, and textile chemistry. Later additions to the curriculum included qualitative and quantitative analysis, physical chemistry, industrial chemistry, chemistry of dyes and dyeing, and chemical microscopy.

Since the incorporation of the college into the University system, curricula have been further expanded to include, for the first time, a chemistry major. In attempting to provide sound professional training within the framework of a liberal arts program, problems typical to most four-year colleges have been faced. In addition to the service courses for other technical majors, a new general education course, taught by the physics and chemistry departments jointly, has been developed during the past two years.

Association with the university minimizes one of the major problems of the small, independent colleges—the budget necessary to provide modern equipment and adequate salaries for the staff.

CHEMISTRY MAJOR PROGRAM

In the liberal arts division of the college, majors are limited to a maximum requirement of 40 semester hours. A uniform program of 33 hours is specified for the chemistry major student. Seven additional hours are chosen from other advanced courses in the field. Eight hours of physics, nine of mathematics, and twelve of German complete the major requirements.

When combined with the general education program required for graduation from the college, the chemistry student is strictly limited in free electives. Two to ten hours, during the four years, is average.

With such rigid specification of program special attention has been given to the content and sequence of chemistry major courses. General, analytical, organic, and physical chemistry form the specified four-year series, with eight semester hours credit for each. Distribution of time between lecture and laboratory varies with the subject.

Each major student, in addition to this basic framework, takes a one-hour course in chemical literature and elects a minimum of seven hours in organic qualitative analysis, advanced inorganic, advanced organic, biochemistry, chemical microscopy, and "independent studies in chemistry."

Strict limitations imposed by number of credit hours allotted to the major are offset by the breadth of knowledge gained from the general education program and instructional advantages inherent in small classes. Within the framework of the course, allowance can be made for the slower, hard-working student, the average, and the superior student. The last of these after completing standard assignments in a course, is encouraged to conduct the more difficult experiments ordinarily encountered in specialized courses in larger institutions. Laboratory time, equipment, assistance from instructors—the prerequisites to this more nearly individualized system of learning—can be found only in smaller colleges.

The general chemistry course is taught primarily for chemistry, physics, and biological science majors.

The New Science Building (Architects' Drawing)

It contains the usual blend of chemical principles, calculations, and descriptive inorganic chemistry. An introduction to qualitative analysis of the cations is taken up in laboratory work during the second semester. By omitting one three-hour laboratory (leaving three lectures and one three-hour laboratory per week), this course has been reduced from ten to eight semester hours during the year. This cut, while originally necessitated to provide additional time for general education courses in the freshman year, has permitted an equalization of the freshman and sophomore courses. In editing the laboratory experiments many were discarded and a few inserted to produce a more constructive and consecutive background for later work.

The additional two hours were added to the second-year course, permitting greater emphasis on calculations dealing with chemical equilibrium. It is felt that this time proves more valuable to the slightly advanced student than to the beginner, since he is better able to grasp the significance of calculations as quantitative applications are made.

The quantitative analysis course now contains two lectures and two laboratories weekly for the year. Representative volumetric and gravimetric determinations are studied during the first semester, with lectures introducing the fundamentals of quantitative analysis and the application of chemical equilibrium calculations. In the second semester gravimetric separations are carried out and some instrumental techniques, including colorimetry, potentiometry, and polarography, are introduced. Lectures deal with advanced topics in quantitative analysis and with principles involved in operation of the instruments and their applications to quantitative determinations.

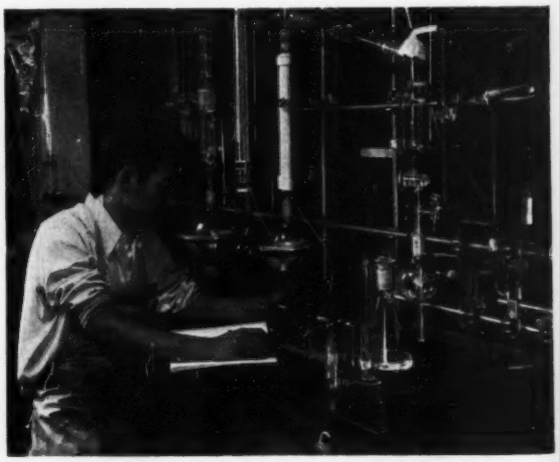
During his first two years the chemistry major student builds a foundation for specialized work by including in his curriculum mathematics through integral calculus and the first-year major course in physics. The first course in German is usually taken during the last half of the sophomore year.

With this background in physics and analytical chemistry, greater emphasis may be placed on physical organic chemistry during the regular third-year organic course. In particular, thorough discussion of reaction mechanisms, dipole moments and acid-base and oxidation-reduction equilibria is possible. Unit operations fundamental to practical organic chemistry are emphasized in the laboratory, rather than mere illustration of representative compounds and synthesis of each class studied.

Training in the use of chemical bibliographical tools is given during the first semester of the junior year with a one-hour course in chemical literature. Both oral and written presentation of technical work are emphasized. A good selection of journals and standard reference works is available through the chemistry library. Beilstein can be used at this point, since the student possesses an adequate background in German.

Physical chemistry is taught as a senior course, with three hours of lecture per week during the year. A separate laboratory course, with two three-hour periods weekly, is taken during the second semester. Equipment is available for nearly all standard physical chemistry laboratory experiments.

In the senior courses the small classes and ready availability of equipment are especially advantageous. Here the student possesses a three-year background in chemistry and allied fields. The slower pupil can be helped individually without retarding the remainder of his class. The average student receives instruction in fundamentals and the use of modern equipment.



Vacuum Fractional Distillation in the Organic Laboratory

His quicker classmate is not held back by a rigid schedule of work or, much worse, bored through lack

of challenge. In the physical chemistry laboratory, for example, while most of the experiments are specified, considerable freedom of choice among outlined work exists in approximately one-quarter of the course.

The independent studies course, while not required, is recommended for nearly all seniors. Originally established as an "honors course," this work has proved of such value to the student that the requirement of a B average is no longer strictly maintained. It is felt that any student to be recommended for graduation should be capable of executing some independent research problem. This viewpoint has been shared by employers of several of our recent graduates, who have felt that success in this course was more significant than the over-all B grade average.

CHEMISTRY FOR NON-MAJOR STUDENTS

A full year course in the physical sciences, carrying three hours credit per semester, has been developed jointly by the physics and chemistry departments to fulfill the need for a general education course.

In developing this course, chemical and physical topics have been interleaved in a logical sequence, rather than separated into "units" of physics or chemistry. This permits a more efficient treatment of such topics as kinetic theory of gases, atomic structure, spectra, and electrochemistry, for example, which must often be presented in an abortive manner through the terminal course in either physics or chemistry, because of lack of background in the other field.

Fundamental principles and concepts of selected subjects in physics and chemistry are stressed, rather than attempting a general survey of physical science. Repeated consideration is given to scientific procedures, as well as to the social significance of advances in fundamental science and technology.

Each lecture section is taught by a member of one department having sufficient experience in the other field to present the entire course. Occasional visiting lecturers are invited for various special topics, but greater unity of viewpoint is gained through a single lecturer for most of the course.

One two-hour laboratory a week accompanies the lectures in this course, with experiments equally divided between physics and chemistry. The chemical experiments are predominantly quantitative, with emphasis placed on the principles derived from them.

Other non-major courses are primarily of the "service" type. Industrial arts and home economics students, needing a thorough understanding of chemical principles together with a survey of descriptive and applied chemistry, enroll in a one-semester introductory chemistry course. A one-semester organic course is also taught, which is taken by home economics students and biological science majors. Both courses include one laboratory weekly.

FACILITIES AND STAFF

Laboratory space is severely limited on the present small campus. Only one laboratory has been added to the department since 1913. In the same period the staff has grown from one to six; the number of courses has been expanded from five to 21.

Construction has begun on a new campus, to be occupied in 1953. In the science building, which will house both physical and biological sciences, the department will occupy an area somewhat greater than that now available, with additional expansion anticipated at an early date. Meanwhile, careful planning for efficient use of space will provide adequate laboratory, lecture room, and office area.

A liberal budget policy during the last few years has permitted acquisition of a Beckman spectrophotometer, Warburg apparatus, semimicro analytical balance, polarograph, and other expensive equipment not ordinarily available to undergraduate students. Such apparatus is selected with the policy that each item is to be used in at least one course during the major sequence. It is available to faculty and advanced students for research use in the remainder of the time. Further purchases at present are limited by space for proper installation more than by lack of funds.

A small, well-equipped shop, maintained jointly by physics and chemistry departments, is available to students and faculty for construction of special apparatus. A part-time mechanician performs the more difficult tasks.

The chemistry library contains a representative selection of periodical sets and reference works which the student is encouraged to use in all major courses. Material not available locally is readily obtained from the Los Angeles or Berkeley campuses through photocopy, microfilm, or interlibrary loan service.

On the new campus arrangement of the library will be based on classification rather than form. For example, all books, reference works, and periodicals which deal with science ("Q" designation in Library of Congress system) will be shelved in one area. Open shelves will be used, thus providing the convenience of departmental libraries without requiring the added expense of maintenance and supervision concomitant to decentralized libraries.

All classes and laboratories are taught by one of the five active staff members. Student assistants are employed for preparation of solutions, setting out chemicals, cleanup work, etc.

Although classes are small, with laboratory sections generally limited to 15, teaching loads are maintained at a relatively low level. Each faculty member has time to carry on a research program and participate in administrative and faculty committees. Research is both expected and encouraged, with funds for special equipment available through the faculty research committee.

In addition to the five regular staff, there is one professor emeritus. Miss Hazel Severy, who retired in 1951 after 37 years with the college, still maintains an active interest in departmental problems, offering the help and counsel of her long career to those of us who are, in comparison, rank novices.

Report of the

NEW ENGLAND ASSOCIATION OF CHEMISTRY TEACHERS

CONSULTING CHEMISTRY AS AN EXAMPLE OF INDUSTRIAL RESEARCH[1]

CORNELIA T. SNELL
Foster D. Snell, Inc., New York, New York

I HAPPEN to be connected with a consulting organization, so I thought you might like to hear about some of the kinds of research problems encountered there. This sort of organization has a broad range of activities, as shown by the following illustrations.

Since I am going to talk about our own group of consulting chemists and chemical engineers I am afraid I shall have to make this more or less personal. Almost 30 years ago my husband—now president of the organization which bears his name—started his career as a consultant while still a graduate student at Columbia University. The business has grown from a one-man, part-time job to one employing over 100 people, who work in some one of a number of different departments, according to their special interests and training. Just as chemistry concerns almost everything from allergy to zirconium, the chemical problems brought to a group of consultants may involve resins or textiles, drugs or cosmetics, construction of chemical plants or destruction of waste materials.

You might like to ask, "What is a consulting chemist?" One definition—probably as good as any—is that he is a member of the chemical profession who, for a fee, attempts to solve chemical problems.

Perhaps you wonder who employs chemical consultants. There are many kinds of clients. Some are large manufacturing firms who maintain their own laboratories but want specialized advice, specialized research, expert testimony in a patent case, or the use of spcialized equipment. Some clients have no laboratories of their own and for them our staff members serve as their chemists. These may be large firms making products of such a nature that their requirements for chemical service are intermittent. Some clients are small firms in chemical manufacture which use our facilities because it is more economical than to maintain a laboratory of their own. For some clients we may take a problem requiring only a week or two of work; for others we carry on research programs which continue over a period of years. Most of our work is in the field of research and development. It is never a question of conducting an investigation at our own expense with the hope of selling the results. A successful consulting laboratory cannot be operated that way. The consultant first gets the client, and incidentally likes to have some assurance that the client will be able to pay his bill. On his part, the consultant must be able to produce results, since only by accomplishment can he establish a desirable professional reputation.

The type of work we do, I think, is best explained in terms of specific examples. I am sure my husband looks back with amusement at some of the simple problems which first came to him. One of his earliest jobs we might call—*à la* Sherlock Holmes—the Case of the Imported Lace. On arrival in New York a shipment of lace was found to have been discolored by water stains. Naturally the question arose as to liability for damage. If the lace had become wet while in the hold of the ship, then the shipping company would be liable for the damage; but if it could be shown that the lace must have been stained before it was loaded onto the ship the shipping firm would bear no responsibility. In order to settle this some of the lace was sent to the laboratory, where it was soaked in water; the aqueous extract was then tested to show the presence or absence of sea-water salts on the lace. Since none were found, this result was reported as reasonable evidence that the damage had not been caused by sea water and that the shipping company was not at fault. An adjustment was made based on this report. Not all consulting work finds such an easy solution.

Sometimes a manufacturer finds himself confronted with a change in his plant or procedure, made necessary by a new law. For example, at one time it was the custom of industrial plants to discharge their waste waters into neighboring streams. In many instances

[1] Presented at the 13th Summer Conference of the NEACT, University of Rhode Island, Kingston, R. I., August 22, 1951.

this caused a change from the naturally clear, sparkling water into a dark-colored, vile-smelling liquid whose odor might carry a mile or so on the wind. Such contaminated waters usually kill vegetation along the banks as well as all fish life. To avoid such a condition many states have passed laws requiring all plants in the state to purify their industrial wastes to meet a definite standard before these are run into streams and rivers. When New Jersey passed such a law many of the dye works located along the Passaic River found that they had a real problem on their hands. Dr. Snell was called in and collaborated with civil engineers in investigating suitable treatments to decolorize and clarify the effluents from these plants. Following the laboratory investigations, several treatment plants were constructed. The precipitated sludge was sent to waste piles while the clarified liquid went into a stream, but no longer with harmful effects.

As another example, our group has been active in the study of synthetic detergents or soapless soaps, such products as you see on the market under the trade names of Tide, Dreft, Breeze, Vel, Glim, Surf, Joy, etc. These contain no soap but do a good cleaning job, and are especially useful in hard water, where soap forms a gummy curd.

In developing the basic material for this general type of product, it is necessary to evaluate removal of soil in the laboratory in order to determine whether one agent is more efficient than another under particular conditions. We have worked out methods of evaluation and have published these, as well as other articles dealing with cleaning problems, in various scientific journals.

In our engineering department, methods and lay-outs are planned on a small pilot-plant scale for later application to full-scale installation.

One client asked us to design a plant in which peanut oil could be made from peanuts or tung oil from tung nuts, depending upon which kind of nut happened to be in plentiful supply at various times of the year. Furthermore, he wanted to build the plant on the Island of Madagascar, an extremely isolated region, as you know, and this meant that additional facilities for generating electric power, process steam, and heating steam had also to be designed. And, finally, machinery for making cans from tin plate had to be installed.

It was a pretty complete job of consulting, but the client got his plant and today not only makes high-grade edible peanut oil, and good-quality tung oil for paints, but the press cakes from the two processes—that is, the solid material that remains after the oil is pressed out—is being used for animal feed from peanuts, and fertilizer from tung nuts.

I mentioned the necessity of meeting the requirements of new state laws. Federal laws affect some manufacturers. For example, manufacturers of cosmetics and other items to be used on the skin must comply with the Pure Food and Drug Act. One of the requirements is that they have no irritant or toxic effect on normal skin. An experimental product is first tested on the skin of animals; for example, a new suntan lotion may be applied first to the shaved skin of rabbits to show possible irritant or harmful effects. If the product passes this test it is then tried on human subjects by means of patch tests on the arms. With a sufficient number of people used, if no more than a few per cent of the subjects show skin irritation the product is considered safe. Physicians and dermatologists often collaborate with us in these skin-test problems. The evidence obtained may be submitted by the client, if required, to the Food and Drug Administration in order to obtain approval of a new product.

Tea tasters have long been recognized as experts in their particular field, but only recently have scientists turned their attention to such matters. It can now frequently be determined not only why one particular product tastes better than another but what the degree of difference is between them. Not everyone can be taught this sort of evaluation but most people can. In our laboratory we have a special tasting panel of 5 to 15 people who have a well-educated sense of taste and can detect very delicate differences in flavor as well as degrees of difference. Many problems can be solved by this method.

For example, a bakery was getting its output back from time to time because the bread, cakes, coffee rings, etc., had a bad taste. There seemed to be no difference in baking procedure or ingredients to account for it. It was discovered that the off-flavor developed only on days when the wind blew from a certain direction—that of a nearby chemical plant. The trained tasting panel found the same unpleasant odor and flavor in the baked goods as that of the chemical made in the neighborhood plant. This illustrates the close cooperation between different departments in a consulting group. Members of the analytical department did the sampling of the atmosphere and the chemical determination, members of the organoleptic panel made the taste evaluation to identify the objectionable material in the baked foods.

Perhaps a typical activity of a consultant—quite different from those just described—is court practice, in both the federal and the lower courts. Judges as a rule prefer that the "expert," the person supplying strictly technical information bearing on the case, be someone not directly connected with either the plaintiff or the defense. That is why this type of work is a natural for the consultant, and no doubt also for members of the teaching profession.

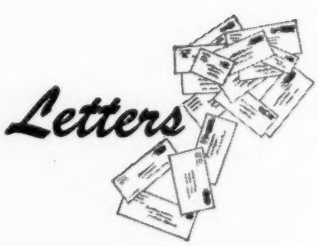
One such case had to do with a blue coating on razor blades. The questions were really two: whether this blue coating actually protected them from rusting and whether two different brands of blue-coated blades were made according to the same patent. Since the blue film was a form of iron oxide only about 720 millimicrons thick, the experimental investigation proved to be quite a problem. The plaintiff in this case was trying to prove that the blue coating did not prevent rusting. In this he was successful but he lost his case on the patent.

THE UNIVERSITY LIBRARIES OF GEORGIA

Sometimes a legal case requires months of research in the laboratory in order to develop the technical background to establish the pertinent facts, even though the trial itself may take only a few days, or weeks at, the most.

Plain common sense has its place in consulting work, as everywhere else. A bottle of liquor in which the whisky had turned black was brought in for examination. We were to find the cause. The analyst started to pour out a sample in order to make a qualitative test for iron. He thought that the liquid probably contained iron tannate, the coloring matter ordinarily present in black ink. When he picked up the bottle and shook it, it rattled. On pouring out the contents, he found an iron nut which had dropped off the filling machine. There was his positive test for iron without the use of reagents.

In conclusion, I like the story about the salesman, who—very discouraged at having to report his inability to make a certain sale—said, "Oh, well, you can lead a horse to water but you can't make him drink." The sales manager replied, "H——, man, your job isn't to lead him to water, your job is to make him thirsty." That is my interpretation of what development work does; it makes you "thirsty" for more knowledge.



To the Editor:

In reading Sister Agnes Ann Green's interesting article on "Microcards" (J. CHEM. EDUC., **28,** 549 (1951)), I am reminded to draw the attention of chemists generally to an urgent problem in this field which has for some time been troubling those of us who are practically engaged in the compiling and editing of compendia.

The problem is an economic one, and rests on the necessity of having a printed presentation of the work in question available before microcards are made. (It would, of course, be possible to have only a single Varityped master copy and a circulation entirely microcard, but this is a novelty, not yet a matter of current practice.)

The use of microcards must, inevitably, decrease the demand for printed copies of the work in question—and whilst, no doubt, the lowered price of the cards will, to some degree, extend the number of those who possess copies, a price differential of \$2200 to \$194 will indubitably divert many sales from the books to the cards.

There comes a point in the publication of books where the number sold just balances the cost of production and distribution; this point represents the present condition of many of the major chemical compendia and treatises, even allowing for the fact that much of the authorship is often a labor of love, paid for at nominal rates. A decrease in sales will either cause a sharp increase in price, or a cessation of publication (probably the latter); nobody will pretend that even a generous royalty on, for example, the \$194 microcard edition of Beilstein is going to make any serious contribution to the cost of preparing that great and valuable work. The problem, therefore, is to ensure that microcard editions make their proper contribution to the economy of script production.

This can only be done by insisting that the microcard publication returns to the original publisher such a royalty as will yield an income equal to that from a similar number of the printed volumes, less that proportion due to the economies of microcards over paper and binding, less some small discount for increased circulation. The production budget of a large work selling at \$2200 appears something like this:

	per copy
Preparation of script (inc. authors' fees)	*500
Setting	*700
Machining	+200
Paper and binding	+200
	\$1600

Items marked * do not vary whether publication takes place as microcards or volumes; items such as + are materially reduced by carding and may be as little as \$100 (no account taken of distribution costs or profit). But even in this way the true production cost (allowing for composition of a microcard edition) cannot be less than \$1300 per copy to which distribution costs must be added. To sell such a set for \$194 is obviously wrong in principle; it may be excellent for old journals where copyright has expired, or where supplies of back numbers are exhausted, but for current compendia it may well kill the goose that lays the golden eggs. *Dictum sapiente sat est.*

G. MALCOLM DYSON

BURTON WALKS
LOUGHBOROUGH, LEICESTERSHIRE
ENGLAND

To the Editor:

I admire Mr. Anthony Standen's contribution "On the horns of the sacred cow" (J. CHEM. EDUC., **28,** 608 (1951)), but would plead for even clearer thinking about "scientific method."

Mr. Standen refuses to define this term in paragraph 1 of his article, but proceeds to use it a hundred times subsequently. It would, perhaps, have been profitable to have told readers in the first instance that the term "scientific method" is used in two quite different senses; to refer to (*a*) the physical methods by which scientists arrive at the data of their subject and (*b*) those philosophical procedures by which the thinking scientist attempts to integrate his observations into the web of knowledge. A textbook of practical organic chemistry is an example of the former, and such a work as "Scientific Method, its Philosophy and Practice" (F. W. Westaway) of the latter. It is confusion between these two ideas that so often misleads young science students.

Under the practical discipline of the laboratory the young chemist learns what it is to be methodical, to be orderly in conducting experiments, neat in arrangement of apparatus, meticulous in taking and recording observations, scrupulously clean lest foreign matter interferes, and of perfect integrity in not allowing preconceived ideas to prejudice observations; in short, to cultivate method in the design and execution of experimental work; and he often thinks of this as "scientific method" (or even "the scientific approach"). In one limited sense this is "scientific method," in that it is the discipline of an investigator in gathering data; but true "scientific method" in the sense that Rayleigh, Armstrong,[1] Westaway, or Alexander Smith[2] meant the term, does not begin until all the former has been completed, and involves the processes of thought and the discipline of philosophy. In these, "good husbandry" is as important as in the experiments of the laboratory; but is far more difficult to attain.

Grote,[3] commenting on Socrates, said:

> In the natural process of growth in the human mind, belief does not follow proof, but springs up apart from and independent of it; an immature intelligence believes first and proves (if, indeed, it ever seeks proof) afterwards.

This is one of the attributes of the mind which makes true scientific method so difficult, and justifies Cicero.[4] "In primis, hominis est propria veri inquisitio atque investigatio."

The analysis of phenomena, the nature of hypotheses, classification, deduction, the use of analogy and inference, the proper place of logic, and the estimation of error and probability in connection with judgment are all features of true scientific method.

This distinction between the two meanings of "scientific method" will serve to dispel Mr. Standen's paradox —that of the general liberal arts student proportionately devoting more attention to true scientific method than the professional science student; for the former takes that which will develop his mental stature and be applicable to all the processes and conditions of life— the latter is stocking his mind with the factual background necessary to a competent technician; the great scientist is the man who does both.

G. MALCOLM DYSON

BURTON WALKS
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[1] ARMSTRONG, H. E., "The Teaching of Scientific Method," **1895.**
[2] SMITH, A., "The Teaching of Chemistry," **1912.**
[3] GROTE, "Plato," Vol. 1, p. 251.
[4] CICERO, *De. offic.*, i, 13.

• ORGANIC SYNTHESES. VOLUME 31

Editor-in-Chief: ***R. S. Schreiber.*** **John Wiley & Sons, Inc., New York, 1951. vi + 122 pp. 1 fig. 15.5 × 23.5 cm. $2.75.**

VOLUME 31 of "Organic Syntheses," like all previous releases, gives the equations for the reactions, procedures for production with accompanying notes, and other methods of production for 42 different organic compounds. The directions have been supplied by 67 different contributors in addition to members of the Editorial Board. The suggested directions have again been further checked or verified by competent referees. Preparations are listed alphabetically under common name and *Chemical Abstracts* indexing name. The subject index comprises all material from Volumes 30 and 31, since it is presumed that all previous preparations, not already listed in Collective Volumes I and II, will eventually appear in Collective Volume III.

This latest addition to this popular series is on a par with all former volumes. The quality, form, appearance, and workmanship are identical to the earlier volumes. This recent release will be welcomed as a "must" by most libraries and organic chemists, both here and abroad. The reviewer as well as many other individuals is still anxiously awaiting the publication of Collective Volume III.

RALPH E. DUNBAR

NORTH DAKOTA STATE COLLEGE
FARGO, NORTH DAKOTA

MELLOR'S MODERN INORGANIC CHEMISTRY

Revised and edited by *G. D. Parkes,* Fellow of Keble College, Oxford, and University Demonstrator and Lecturer in Chemistry. Fourth edition. Longmans Green and Co., New York, 1951. xxi + 967 pp. Illustrated. 73 tables. 22.5 × 15 cm. $5.

THIS book is designed as a text for the first course in chemistry for college students in England. As might be expected, it is at a somewhat higher level and contains more material than most comparable American texts, but interested American freshmen could use it without undue difficulty. As is usual in English textbooks, the literary style and choice of words are excellent.

This revision is in reality the tenth edition of a text which has been highly successful and widely used in England and other parts of the world since 1912. In format and appearance it closely resembles the ninth edition (1939), but an examination of the contents shows that considerable change has been made. In spite of this, the book will seem "old fashioned" to many American readers, for in selection of topics and emphasis upon them it resembles American texts of 20 years ago. The historical approach is used throughout, with the older views often given greater emphasis than the newer; thus, the original Arrhenius theory and the short form of the periodic table are used almost to the exclusion of more recent theories. While descriptive chemistry is stressed, a considerable amount of space is devoted to classical physical chemistry: *e. g.*, phase rule, catalysis, conductivity, and the order of reactions. Atomic structures are discussed, but little use is made of them in explaining or correlating the properties of the elements; molecular structures are rarely mentioned. It is significant that Chapter 9 (The Constitution of Matter) is all in fine print.

Numerous examples can be cited to indicate that this book has not been adequately brought up to date; of the transuranium elements only neptunium and plutonium are mentioned; the discussion of element 87 ends with the Noddacks in 1925; the possible existence of a second "rare earth" series is not discussed; the silicone resins are omitted; sodium polymetaphosphate is described as the hexametaphosphate. The statement (page 289) that "non-metals for the most part do not react with water" is quite erroneous, as such reaction is characteristic of all of the nonmetals except oxygen, nitrogen, and the noble gases. The statement that lead exhibits trivalency (page 749) is also incorrect; X-ray studies have shown that the oxide Pb_2O_3 contains equal numbers of Pb(II) and Pb(IV) atoms. Baker's work on intensive drying, which most chemists have now rejected, is mentioned repeatedly.

On the other hand, the chapters on the metals and on the structure of silicates are excellent, as is the discussion of chemical equilibrium and velocity of reactions.

Mellor's approach to inorganic chemistry is so different from that commonly used in America that few teachers in this country will wish to adopt his book as a text. The fact that the point of view is different, however, gives the book some value as a reference text, for the teacher and the student.

UNIVERSITY OF ILLINOIS
URBANA, ILLINOIS

JOHN C. BAILAR, JR.

PREPARATION OF ORGANIC INTERMEDIATES

David Allen Shirley, Associate Professor of Chemistry, Tulane University of Louisiana. John Wiley & Sons, Inc., New York, 1951. x + 328 pp. 15 × 24 cm. $6.

THE purpose of the book is to provide the laboratory worker with specific preparative directions for relatively simple compounds which are unavailable commercially, or are expensive enough to justify their preparation by the worker.

The author states in the preface that the usefulness of the book will depend primarily on the compounds selected for inclusion. The more than 500 compounds were chosen on the following basis: (1) not available commercially, or, if available, relatively expensive; (2) the compound has not been described in "Organic Syntheses" through Volume 28; and (3) "the compound is one whose structure is simple and contains reactive functional groups which make it useful as an intermediate, or its preparation involves a generally useful type of organic reaction and the directions may be applied to the preparation of related compounds."

All of the starting materials meet one of the following criteria: (1) commercial availability at low cost; (2) described in "Organic Syntheses"; or (3) described elsewhere in the book.

The compounds are arranged in alphabetical order and the following information is supplied for each compound: the equation for the reaction, the reference to the original literature, a condensed version of the original procedure, and, in some cases, special precautions to be observed in carrying out the procedure. There is also a molecular-formula index, type-of-reaction index, and general index.

The procedures were chosen by searching the literature for all the methods available, and then, after a careful and critical examination of these, selecting the one which in the author's opinion has a high probability of giving the indicated results. As Professor Shirley also states, difficulty is sometimes experienced in attempting to reproduce experimental procedures taken from the original literature. Unlike the procedures in "Organic Syntheses," the ones in this book *have not* been checked by independent investigators. Also, no references are given to any alternative procedures which may be available. In the reviewer's opinion, these two features seriously limit the usefulness of the book.

The binding is attractive and the format, patterned after that of "Organic Syntheses," meets the high standards one is accustomed to in Wiley publications.

HAROLD R. NACE

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RADIATION MONITORING IN ATOMIC DEFENSE

Dwight E. Gray, Chief, Navy Research Section, Library of Congress, and _John H. Martens,_ Technical Information Service, Atomic Energy Commission. D. Van Nostrand Co., Inc., New York, 1951. iv + 122 pp. 20 figs. 14 × 21 cm. $2.

THE task of training civil defense workers has been made more complex than it was during World War II by the introduction of "atomic" warfare with its associated hazard of nuclear radiation. Recognizing the need for a text dealing with this new hazard, the authors of this book have presented for the general reader, as well as the civil defense worker, a discussion of the principles and practices of radiation detection and monitoring.

The seven chapters in the first part of the book are devoted to background information. This includes the following topics: nature of chemical elements and isotopes, nuclear energy, properties and detection of nuclear radiation, fission, nuclear explosions, radiation hazards associated with nuclear explosions, and protective measures.

In the reviewer's opinion, two of the specific topics covered as background information should have been discussed in greater detail with illustrations. These are the half-life of a radioactive substance and the roentgen as a unit of radiation dosage.

In the eight chapters of the second part of the book instruments and equipment for radiological monitoring are described. Specific instructions are given for the operation, calibration, use, and maintenance of several commercially available survey meters. Ionization-type pocket chambers and dosimeters and film badge dosimeters are also described. As pointed out by the authors, new instruments are being developed and may replace those which are considered in detail in the text. It is likely, however, that new instruments will differ in size and cost rather than principle of operation.

There are two appendixes. The first is devoted to a description of standard sources of radiation for calibration of equip-

ment; the second consists of a bibliography with comments on the publications listed.

In general the authors have done well in their brief presentation of the material. The book is definitely useful for the training of personnel for radiological defense.

HERBERT M. CLARK

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• FUNDAMENTAL PRINCIPLES OF PHYSICAL CHEMISTRY

Carl F. Prutton, Vice-President and Director of Operations, Mathieson Chemical Corp., and *Samuel H. Maron,* Professor of Physical Chemistry, Case Institute of Technology. Second edition. The Macmillan Co., New York, 1951. x + 803 pp. 173 figs. 127 tables. 15 × 22 cm. $5.75.

SINCE the appearance of the first edition of this text in 1944, it has been widely adopted. Except for one completely new chapter, and a substantial increase in the number of problems, this revised edition differs very little from its predecessor. Addition of a few paragraphs here and there has been offset by deletion of others, so that, except for the added chapter, the number of pages is almost unchanged. Since there has been very little rewriting, most of this edition is identical with the last.

The discussion of crystal structure has been improved by the addition of three paragraphs on space lattices and space groups. The treatment of adsorption isotherms has been slightly expanded, and a section on the Donnan equilibrium has been added. This last is a complex subject, and as presented here (as in most such tests) it is oversimplified. In the reviewer's opinion, unless a great deal more space can be devoted to it, including application to actual systems, it might as well be omitted entirely. The chapter on thermochemistry has been improved by the addition of a discussion of differential heats of solution and dilution.

Several changes have been made in the chapters dealing with atomic structure. Discussion of mass defect has been somewhat expanded, and the description of the nucleus of the atom has been improved—the earlier edition talked about electrons in the nucleus. A paragraph on the generation of neutrons has been added, and the brief section on nuclear fission has been brought up to date.

The major change in the book is a new chapter on photochemistry. Like the rest of the book, this chapter is clear, well written, and teachable.

WILLIAM E. CADBURY, JR.

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• HERBERT H. DOW: PIONEER IN CREATIVE CHEMISTRY

Murray Campbell and *Harrison Hatton.* Appleton-Century-Crofts, Inc., New York, 1951. xi + 168 pp. Illustrated. 18.5 × 26.5 cm. $3.50.

BRINE was considered merely salty water with few chemical potentials until young Herbert H. Dow made the study of this solution his life's interest. In 1889, after graduating from the Case School of Applied Science, Dow became Professor of Chemistry and Toxicology at the Huron Street Hospital College in Cleveland. This position gave him spare time for analyzing and experimenting with brine. His first attempt to extract bromine from brine on a commercial basis took place near Canton, Ohio. Shortly thereafter, Dow went to Midland and under various names the eventual Dow Chemical Company took shape.

This attractive book by Campbell and Hatton chronicles the successes and disillusionments which constantly beset Dow. Several interesting sections discuss his relations with the boards of directors and financial backers of the various companies where Dow acted as founder and general manager. The difficulties of raising money for expanding production and developing of new products are vividly told.

Herbert Dow, by his willingness to try anything that should work and make it work, established a pattern that catalyzed the rapid growth of the Dow Chemical Company. Dow's specific contributions to the growth of chemical manufacture in this country include: the introduction of cheap phenol; the creation of a dependable domestic source of metallic magnesium; the battles won over foreign monopolies in chemicals like bleach, indigos, the salicylates; the development of carbon tetrachloride as a raw material for other chemicals and as a volume chemical in itself; the development of cheap chlorine and its wide use as a processing material; the continuing search for new uses for bromine; and so on. Along with all these developments, the Dow Chemical Company grew and became a leader in the industry by Dow's continuous striving for diversifying products and improving the efficiency of chemical processing.

Herbert Dow's contributions of a general nature are his pioneering and improving of the continuous process which is automatically controlled and operated; the ceaseless search for new products and ways of making them; the exploitation of unwanted by-products; the constant effort to make products more cheaply; the driving interest in using cheap raw materials, such as brine; the commercial importance of research; and, perhaps above all, the sheer creativeness of Dow's industrial attitude.

This book is really the biography of an outstanding chemical company whose life was sparked and nurtured continuously by Herbert Dow and the fine group of hand-picked men which surrounded him at all stages of the company's development. Although the story ends with Dow's death in 1930, his spirit of enterprise continues as the greatest single motive force in the company today. Anyone looking for a chronicle of intelligent capitalism and the American way of life will find this book rich and rewarding.

FREDERICK G. SAWYER

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• SURFACE ACTIVITY

J. L. Moilliet and *B. Collie,* D. Van Nostrand Co., Inc., New York, 1951. x + 379 pp. 22 figs. 6 tables. 15.5 × 25.5 cm. $12.50.

THIS book provides an excellent treatment of surface-active agents and surface activity from the point of view of the fundamental physical chemistry involved. It is divided into three parts: the first, of about 150 pages, physicochemical principles; the second, of about 100 pages, deals with technical applications; and the third, of about 120 pages, presents the chemical constitution of synthetic surfactants.

In Part I theories and substantiating data dealing with the nature and properties of surfactants are summarized. For example, the nature of micelles and the conflicting theories as to their probable size and structure are treated in some detail. Many references placed at the bottom of the page—both in this part and throughout the book—refer the reader to the original articles. This generous use of references permits condensation of material with which the reader may be assumed to have some familiarity, or if not, which he can readily look up.

The technical applications in Part II furnish good general discussions of the factors involved in surface activity, including wetting-out and waterproofing, emulsification, dispersion and deflocculation, solubilization, etc. Again, emphasis is on the physicochemical principles involved. Some examples of commercial applications are given. These tend to deal with industrial processing of textiles, although other types of applications are mentioned. Uses of surfactants in the manufacture of cosmetics are not discussed. The building of syndets for household use is not treated.

In Part III the surfactants are grouped by general classifica-

tions. The constitution of the various types is discussed, with chemical formulas and with examples in many instances of trade-name products. These draw on both American and European products but are meant to be illustrative rather than inclusive. The raw materials used and methods of manufacture developed by a number of companies are referred to.

The book deals with a highly complicated subject and naturally does not cover all phases and applications. It should be of value to all those interested in the technical aspects of surfactants.

CORNELIA T. SNELL

FOSTER D. SNELL, INC.
NEW YORK, NEW YORK

● CHEMISTRY OF WOOD

Erik Hägglund, Swedish Forest Products Research Laboratory Stockholm. Academic Press, Inc., New York, 1951. x + 631 pp. 86 figs. 16 × 23.5 cm. $13.50.

A BOOK reviewer always starts with the preface; and, indeed, he may lean as heavily on those pages numbered with Roman numerals at the beginning as he does on the bulk of the writing itself in a much larger total of Arabically numbered pages. The old Latin books started with an "apologia" but no apology is necessary here. However, Dr. Hägglund begins his preface: "This book is a new, revised English edition of 'Holzchemie,' the first edition of which was published in 1928. Since the second German edition was published in 1939 great strides have been made in the various fields of wood chemistry. The results of this new research have greatly widened our knowledge and have necessitated a revision of many concepts held formerly. A comparison between this book and the previous edition clearly illustrates this fact."

One additional interesting comparison is that, whereas before World War II the text was published exclusively in German in two editions, the postwar results have been printed in excellent English by a press in Sweden. The relative growth of importance of the English language in the field of technology of wood is thus indicated.

No one is better fitted nor more competent to write a book on this subject matter than the author, with his outstanding years of experience and direction in the Swedish Forest Products Research Laboratory, one of the great research institutions of the world. Fortunately also, while the title of the book is "Chemistry" the subject matter of the book does not have the pure scientific approach that might thereby be expected; it is almost entirely written from the aspect of the industrial utilization and processing of wood along the many avenues which has made this material such an important raw material in the last century from the chemical and semichemical standpoint. Throughout the entire history of mankind's manufactures, or his conversion of raw materials into more valuable finished products, wood has been the most frequent and versatile raw material for mechanical conversion; and in these more recent years the chemical approach has been an important additional method of utilizing wood in an even larger number of ways, and of forming even more valuable finished products. It is this scientific background of this multi-billion-dollar industry which is described.

The outline of the field of interest, as indicated by the table of contents and by the subject material, follows a perfectly scientific exposition of the chemistry, the properties, the reactions with reagents, and the chemical processing involved in the conversion of woody substance to other products. Following the first four chapters (389 pages) on the physical and chemical properties of wood (considering in order the fundamental components of wood) there follows one chapter on the saccharification of wood, *i. e.*, the decomposition and dissolution of the cellulose by hydrolysis with acids, while the lignin is relatively nonreacted. The next four chapters (116 pages) are concerned primarily with the pulping of wood and the chemical dissolution therefrom of the lignin by all of the different methods which have been proposed and which leave the cellulose relatively nonreacted.

Chapter 9 relates to wood carbonization; Chapter 10 covers caustic fusion and miscellaneous other treatments with alkalies, tars, or phenols; and in all of these both cellulose and lignin are entirely reduced—in the one case to give solid charcoal, and in the other liquids or solutions. Finally there are two chapters, 11 and 12, on the behavior of wood during storage and the natural decomposition of wood by enzymes and microorganisms to give humus, coal, etc.

One of the best features of the book is the extremely complete bibliography of references to workers in all countries and in all languages relating to this field. Using Sweden as a center, Professor Hägglund is in an excellent position to survey the contributions to this field of all countries of the world, and he appears unbiased in his inclusion or exclusion of material covered. Most of the important American references are covered.

This treatise is primarily concerned with industrial chemistry rather than technology and there are no figures, flowsheets, or photographs of processing operations. The 86 figures are somewhat fewer than a well-illustrated book on industrial chemistry would require, but these are, however, excellent graphs of physical data representation and line drawings and photographs of the fiber structures and related mechanical aspects of molecular and component organization.

The excellent and scientific approach to the coverage of the field is shown by the 50 pages of author index and 16 pages of subject index, giving a comprehensive and carefully organized background for ready reference to almost any subject point or publication in the field. In addition there is a bibliography at the end of each chapter or section.

As should be expected of a book on wood which has been printed in Sweden, the paper, type, format, and binding are excellent.

While a treatise rather than a textbook, this will be used by graduate students (and by everyone else interested in the field) as required reading, and as a ready point of departure for the tremendously voluminous literature in this field.

DONALD F. OTHMER

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● PREPARATION, PROPERTIES, AND TECHNOLOGY OF FLUORINE AND ORGANIC FLUORO COMPOUNDS

Edited by _Charles Slesser_ and _Stuart R. Schram,_ Director and Consultant, respectively, Division of Technical Information and Declassification Service, New York Operations Office. McGraw-Hill Book Co., Inc., New York, 1951. xxiii + 868 pp. Illustrated. 16 × 23.5 cm. $10.50.

THIS book is one volume of a series prepared as a record of work done under the Manhattan Project and the Atomic Energy Commission. It is divided into five parts, containing a total of 40 chapters written by 60 different authors, and the editors' names do not appear in the list of authors. The work was done at a number of locations, and the chapters are more or less the summary reports of the different research and development teams. The reader can therefore expect to find considerable variation in style and quality of the different chapters, as well as a considerable amount of duplication. The book is an historical record of development activities done under the stress of war and is an account of technical accomplishments of high order that at their start appeared to be on the verge of the impossible. That much of this is not suitable for industrial use, or has subsequently been made obsolete, does not detract from its greatness or the value of having it available in a written record.

Despite the unavoidable duplications in Parts I and II, they contain much useful information about the preparation and handling of elementary fluorine on an industrial scale. A number of chapters in the other parts of the volume have considerable merit as, for example, chapters 32 and 39.

The object of the development activities was the preparation of a variety of fluorocarbons having different physical properties

for a number of different uses, in connection with the separation of the uranium isotopes by the diffusion process employing UF_6. The cohesive theme of the book is the story of the various attempted paths to this end. As the only published method of making fluorocarbons at that time used elementary fluorine, and as this was needed for other purposes, large-scale equipment was developed for what previously was only a laboratory preparation. Part I contains reports of this work. Part II contains technical information from the various research teams relative to large-scale handling of elementary fluorine. As the production of fluorine proved to be technically difficult, means were sought for fluorocarbon production requiring less of the free element. Part III reports the institutional and industrial development of the employment of the Swarts reaction, or its modification using hydrogen fluoride, for obtaining fluorine-containing compounds from chlorine-containing ones. Part IV contains accounts of the various successful processes for the production of fluorocarbons with their advantages and disadvantages. As these were technical masterpieces, the reports are of considerable historical value, despite the fact that they probably will be of little value as directions for future processing. Part V contains, in addition to the preparation and properties of chlorotrifluoroethylene polymer, an account of the excellent work of Dr. W. I. Miller, Jr., on the polymerization of olefinic fluorocarbons. In Part VI are collected reports of miscellaneous investigations related to the general theme of fluorocarbon production.

J. H. SIMONS

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• PHYSICAL CHEMISTRY OF LUBRICATING OILS

A. Bondi, **Shell Development Co., Emeryville, California, Reinhold Publishing Corp., New York, 1951. vii + 380 pp. 128 figs. 16 × 23.5 cm. $10.**

The fundamental principles of physical chemistry have been applied in this book to the heavy hydrocarbons in lubricating oils. It does not deal with the refining processes, but with the properties of the lubricating oil. Those principles applied are best indicated by the titles of the eight chapters: P-V-T Properties, Rheology, Surface Phenomena, Optical and Electrical Properties, the Hydrocarbon Type Analysis of Lubricating Oils, Phase Equilibria, Reaction Kinetics, Synthetic Lubricants.

The author has made an authoritative survey of the application of those physical chemical principles. Each chapter is thoroughly documented with references to the literature. Physical chemists working with other viscous materials will find much in this book of interest to them.

KENNETH A. KOBE

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• COMBUSTION, FLAMES AND EXPLOSIONS OF GASES

Bernard Lewis **and** ***Guenther von Elbe,*** **Physical Chemists, Explosives and Physical Sciences Division, U. S. Bureau of Mines, Pittsburgh, Pa., Academic Press, Inc., New York, 1951. xix + 795 pp. 355 figs. 16 × 24 cm. $13.50.**

This book by these well-known authors is a welcome addition to the rapidly growing literature in the field of combustion and explosions. As the authors correctly point out, it is not to be considered a new edition of the book they published with the same title in 1938. While comparisons between the two books are inevitable, the current book is considerably more comprehensive in its scope and several times the size of the earlier work.

The book is divided into four parts. This first part, entitled "Chemistry and Kinetics of the Reactions between Fuel Gases and Oxygen," is concerned with the oxidation of hydrogen, carbon monoxide, and hydrocarbon. This portion, which comprises one-fourth of the book, may seem unduly long; however, it contains a great deal of detail and effectively summarizes the work of many people over several decades. Chain mechanisms are discussed in considerable detail. The attempt to coordinate the various reactions of the several mechanisms in this section by consecutive numbering is commendable, but unfortunately it becomes somewhat cumbersome after more than 100 pages with almost as many numbered reactions. This part of the book also includes a summary of the basic equations and concepts of kinetics and chain reactions, a discussion of cool flames and two-stage ignition, as well as a short section on engine knock.

About half the book is concerned with "Flame Propagation" in Part II, which includes subject matter ranging from nonturbulent combustion waves to gaseous detonations. The authors have nicely interwoven the discussions of experimental methods and qualitative pictures with extensive theoretical presentation. The development, propagation, and stabilization of flames under turbulent and nonturbulent conditions are discussed in detail. The hydrodynamic theory of combustion and detonation, while not always developed completely from first principles, is in general presented in a form useful to the average research worker.

Part III is entitled the "State of the Burned Gas" and is concerned with equation-of-state information as available from band spectroscopy or from measurements made during explosion or burning processes. The measurement of temperature—principally by the line reversal method—is discussed.

The last part of the book, "Problems in Technical Combustion Processes," is a very short section on some of the practical aspects of combustion. While detailed discussion is obviously outside the scope of this book, there is included an outline of the Otto, Diesel, gas turbine, and turbo-jet engine cycles, and their combustion processes.

Valuable data on equilibrium constants, heats of combustion, limits of inflammability, and flame temperatures have been included in the appendixes.

In general the book is well written, the printing good, and the photographic reproductions (particularly those of turbulent flames) are excellent. While it is doubtful if this work will be selected very often as a classroom textbook, it will undoubtedly serve as a very valuable source book to the teacher, graduate student, research worker, and combustion engineer.

PAUL M. FYE

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• LEHRBUCH DER ORGANISCHEN CHEMIE

A. F. Holleman **and** ***Friedrich Richter.*** **Revised by** ***F. Richter,*** **Editor of Beilstein's "Handbuch der organischen Chemie," Frankfurt a. Main. 27th and 28th edition. Walter de Gruyter and Co., Berlin, 1951. xii + 526 pp. 97 figs. 28 tables. 18 × 24 cm. DM 24.**

The continued popularity of this 53-year-old textbook seems to be aptly demonstrated by a remark made by F. Richter, editor of the book since 1930, in the preface of the latest edition. He states that the rapid sale of the 26th edition necessitated the printing of a new edition within the space of a year. This latest edition has been "replenished" after a careful review of the progress of the literature of organic chemistry. Probably by necessity, the selection of new material has been somewhat arbitrary. The book has been printed with meticulous care and the format is quite attractive.

The arrangement of the text is in the classical German tradition. There is much descriptive material, and many reactions are not symbolized but discussed in the running text. There is no formal use of subheadings, such as reactions of, preparations of, etc. Abundant cross references occur throughout the text. Aliphatic and aromatic compounds are discussed separately. Approximately 270 pages are devoted to acyclic compounds, about 120 to the strictly aromatic compounds, about 60 to the

heterocyclic compounds, and about 32 to the hydroaromatic compounds and the steroids. This allotment of space is indicative of the author's over-all treatment of the subject matter.

The rather inconsistent use of symbols may annoy some readers. In the graphical formulas of the olefins the point of unsaturation is often indicated by two dots, and in other cases by the so-called double bond. A similar pattern obtains in the discussion of the acetylenic compounds. The proton is usually designated in the conventional manner. However, on page 77 a superscript dot is used instead of a positive sign. Radicals are often written by placing a dash perpendicular to the symbol, while at other times nothing is next to the symbol. On page 115, in the symbolic representation of Kharasch's abnormal addition to olefins, the unpaired electrons are indicated by an X. This procedure is used also on pages 413 and 415. However, since the format of the structural formulas is similar to that in Beilstein most readers should have few difficulties.

Some readers may consider certain sections to be rather weak—for instance, the alcohols, the phenols, the aromatic nitro compounds, the carbohydrates, and the index. The structural formulas of the disaccharides are treated rather casually. In the case of cellobiose and sucrose the stereochemistry within the monosaccharide fragments is ignored completely.

Because the text is in German it will be used infrequently as a textbook in this country. However, many teachers and advanced students may wish, and perhaps should have, a copy in their libraries. The book offers an excellent opportunity for students to gain facility in the use of German, since the text contains essentially familiar material.

DONALD C. GREGG

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● SELECTIVE TOXICITY

Adrien Albert, **Professor of Medical Chemistry, The Australian National University, and Honorary Lecturer in the Department of Chemistry, University College, London. Methuen & Co., Ltd., London; John Wiley & Sons, Inc., New York, 1951. xiii + 228 pp. 5 plates. 31 figs. 27 tables. 11 × 17.5 cm. $1.75.**

IN THIS book the author has presented an interesting and well written discussion of a subject that has long needed integration.

The book is based on postgraduate lectures delivered to research workers and students in biochemistry, medicine, chemistry, veterinary science, and agriculture. It should appeal not only to these but also to the general reader in the more advanced disciplines in the physical and natural sciences.

Selective toxicity is defined as the toxicity possessed by certain agents which injure some kinds of cells but not others. Hence it is concerned with drugs and weed-killers and insecticides. The emphasis in this book is not so much on the agents themselves as on the physical and chemical means whereby these agents effect their activity. Much information, therefore, which belonged formerly to technology has been rigorously examined and the underlying principles involved have been brought into focus.

In his opening pages the author gives an account of the accomplishments of selective toxicity and the scientific basis for selective toxicity in terms of biology and the physical sciences. Selective toxicity is then discussed in relation to physical and chemical properties with special reference to: (*a*) the covalent bond, as in the arsenicals and mercurials, (*b*) ionization, as in the acridines and other katonic antibacterials, (*c*) chelation, as in folic acid, 8-hydroxyquinoline and other carriers and inactivators of trace metals. The author's activity in these last two fields permits him to write with considerable authority and meaningfulness. Indeed, throughout the book he has succeeded in bringing appropriate reference from the current literature.

In a later section Dr. Albert discusses chemotherapy as a particular branch of selective toxicity tracing its fundamental principles and dealing in detail with such topics as sulfa drugs, antibiotics, drug resistance, and therapeutic interference.

An excellent bibliography is appended.

LIONEL JOSEPH

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● DIELECTRIC BREAKDOWN OF SOLIDS

S. Whitehead, **Director of Research, The Electrical Research Association, England. Oxford University Press, London, 1951. xv + 271 pp. 75 figs. 14.5 × 22.5 cm. $5.**

A KNOWLEDGE of the factors which influence dielectric breakdown is of fundamental importance in the electrical industries. This book presents selected experimental results illustrating performance of dielectrics under varying conditions. The data are chosen primarily, however, to illustrate and test the more successful theories of breakdown. Very high potential gradients, of the order of a million volts per centimeter, cause intrinsic breakdown. Breakdown occurs when electrons in the solid acquire energy more rapidly from the field than they loose it to the solid lattice. A theory due to Fröhlich has been comparatively successful in interpreting this intrinsic breakdown. Again, a solid dielectric often fails because the applied electric field generates currents which heat the solid faster than this heat can be carried away without reaching destructive temperatures. The consequences of this for breakdown are explored both theoretically and experimentally. Other chapters discuss breakdown caused by discharges and by electrochemical deterioration, while the final chapter treats dielectric breakdown in practice.

The book is a valuable advance in the exposition of the principles involved in dielectric breakdown. It should be read by both the fundamental and applied scientists interested in solid state change. Much of the book is nonmathematical.

HENRY EYRING

UNIVERSITY OF UTAH
SALT LAKE CITY, UTAH

● QUANTUM MECHANICS OF PARTICLES AND WAVE FIELDS

Arthur March, **Professor of Theoretical Physics, University of Innsbruck, Austria. John Wiley & Sons, Inc., New York, 1951. x + 292 pp. 5 figs. 15.5 × 24 cm. $5.50.**

THIS book presents the fundamental theory of quantum mechanics with especial emphasis on relativistic theory. In the last chapter Dr. March develops his own idea for removing the difficulty in the usual theory which associates an infinite energy with any enclosure. Thus, in a rectangular box all frequencies are allowed for which an integral number of wave lengths correspond to the box length. The associated half quanta thus constitute an infinite zero point energy. The author eliminates the high frequencies by assuming "it is, on principle, impossible to invent an experiment of any kind that will permit a distinction between the positions of two particles at rest the distance of which is below a certain limit l_0." This assumption removes part of the difficulties only but it is introduced and developed in an illuminating manner. Dirac's relativistic equation and his hole theory are developed in an instructive way.

The book is always clear and will be especially valuable in physics courses devoted to a careful examination of basic principles.

HENRY EYRING

UNIVERSITY OF UTAH
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Some Modern Texts for Chemistry Students

A COURSE IN COLLEGE CHEMISTRY

by V. R. Damerell

Designed especially for the terminal course in first year college chemistry, this book firmly ties abstract chemical concepts to concrete everyday examples and thus avoids confusing the student with a melange of technical terminology and complicated theory. The author presents the chemistry of groups of substances as they occur in nature and in daily life, the progression being chiefly in order of increasing molecular complexity. *Ready in Spring.*

TEXTBOOK OF QUANTITATIVE INORGANIC CHEMISTRY, *3rd Edition*

by I. M. Kolthoff & E. B. Sandell

In revision practically all the theoretical chapters have been thoroughly rewritten. The chapter on "Separations" has been entirely rewritten and is a unique feature of the book. A comprehensive review of electrical methods of analysis is given, and modern developments in the fields of complex formation, organic reagents, etc., are presented in a fundamental and concise way. In the part dealing with *Physical Methods of Analysis* (previously called *Physico-Chemical Methods of Analysis*) the flame photometer and use of radioactive methods in analytical chemistry are described. *Ready in the Spring.*

THE CHEMISTRY OF ORGANIC COMPOUNDS, *4th Edition*

by James B. Conant & A. H. Blatt

An essentially complete revision of the text is represented in this new fourth edition. The changes in the arrangement of the chapters are minor. In the new edition the emphasis on the essential facts of organic chemistry and the empirical generalizations that serve to correlate so many of these facts have been retained. Electronic interpretations of the course of organic reactions have been used to a greater extent than in the preceding edition but are still used sparingly. The most important new material is a discussion of the principal types of mechanisms by which organic reactions take place. *Ready in the Spring.*

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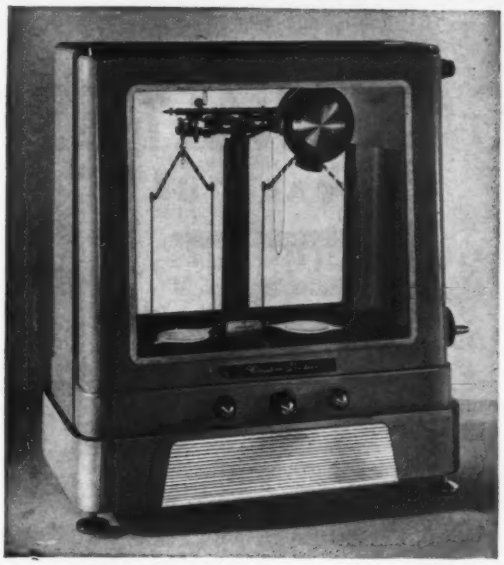

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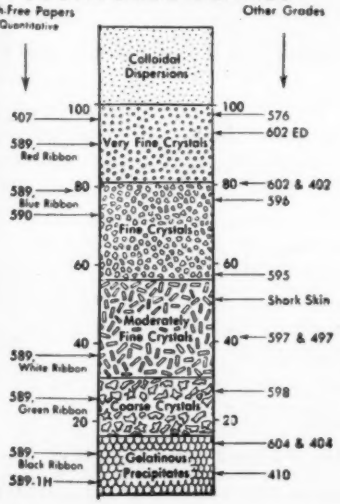

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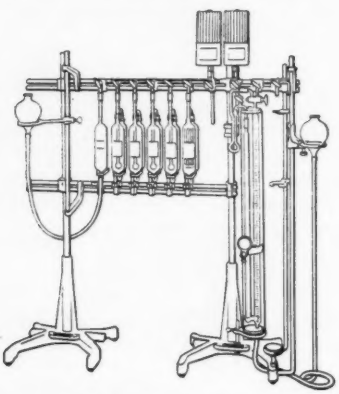

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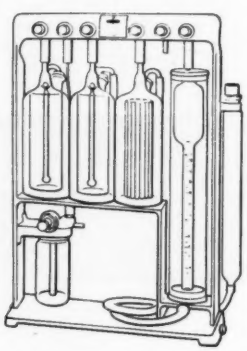

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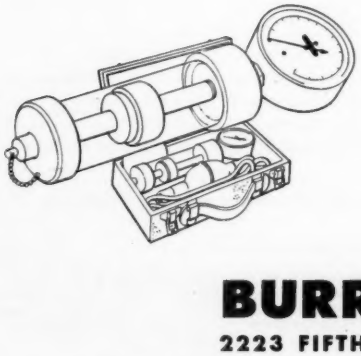

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The Ohaus Scale Corporation of Union, New Jersey, announces the incorporation of self-aligning bearings in its heavy-duty solution balances.

According to the manufacturer this feature will improve the sensitivity by reducing friction and will materially increase the life of the balance by assuring full contact with the knife edges at all times, thus distributing the wear evenly over the entire knife edge.

A unique system of hardened steel check levers permits a sensitivity of one gram under loads up to its capacity of 20 kilograms. This large capacity along with its fine sensitivity makes the scale useful for many purposes in research, agricultural and industrial laboratories.

The balance can be supplied with a stainless-steel platform or with subject box for weighing of animals. Models are available in either the metric or avoirdupois standards or a combination of both.

For further information and free literature, please address inquiries to Ohaus Scale Corporation, 1050 Commerce Ave., Union, New Jersey.

Deeminizer Folder

A four-page folder describing the Deeminizer is available from Crystal Research Laboratories, Inc., 29 Allyn Street, Hartford 3, Conn. This inexpensive ion exchange unit is said to produce mineral-free water of high purity. It is capable of producing up to five gallons of chemically pure water per hour.

The unit features a built-in purity indicating meter which gives the user an accurate check on the purity of the water at all times.

New "Waco Catalyst"

"WACO Catalyst," Volume 9CE, has just been published by Wilkens Anderson Company, 4525 W. Division Street, Chicago 51, Ill. The current issue is virtually a complete catalog of Coleman instruments showing their line of titrators, PH meters, spectrophotometers, etc. It also illustrates and describes a number of Waco accessories specially designed to increase the range and applications of the Coleman spectrophotometer.

Copies of "WACO Catalyst," number 9CE, are available on request.

Freeze-drying Unit

A small freeze-drying unit for general research and laboratory work has been introduced by the F. J. Stokes Machine Co., 5500 Tabor Road, Philadelphia 20, Pa. Suitable for drying cultures and other products in small volume, it will dry up to 24 containers simultaneously. Large containers up to 250 ml. total capacity can be handled.

The unit consists of a tank and chrome-plated manifold with 24 outlets, and a vacuum-insulated condenser bath. It is available with or without vacuum pump and McLeod gage.

New X-Ray Diffraction and Spectrometer Catalogue

A new 60-page catalogue titled "X-ray Diffraction and Geiger-Counter X-Ray Spectrometric Equipment" has been released recently by the Research & Control Instruments Division, North American Philips Company, Inc., 750 South Fulton Avenue, Mount Vernon, N. Y.

In addition to X-ray diffraction, spectrometry, and fluorescence analysis, the catalogue covers such components and accessories as tubes, rectifiers, and cameras. It also has sections on camera mounting brackets, film illuminators and measuring devices, and monochromators.

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References

(1) Ind. Eng. Chem. 25-653 (June, 1933)
(2) Ind. Eng. Chem. 25-1112 (Oct., 1933)
(3) National Bureau of Standards Journal of Research 12-241 (Feb. 1934, R.P. No. 649)

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Considerable space in the new catalogue is devoted to the Philips Electron Microscope. Other data are included on such items as the flat specimen spinner, counting rate computer, special holders, milliampere stabilizer, Weissenberg and Geiger counter, Weissenberg cameras, crystal analysis equipment, and thickness plating gauge.

Information is also provided on applications for X-ray instruments along with typical charts which show how specimens differ when analyzed by this powerful laboratory and production control equipment.

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To provide improved heat uniformity throughout and improved heat transfer between heater bank and load in the oven, Blue M Electric Company has developed the new "Con-Wate" mechanical convection oven.

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Further information may be had from the manufacturer, Blue M Electric Company, 306-308 West 69th Street, Chicago 21, Illinois.

New Literature

A new, 20-page manual outlining methods for the analysis of copper and lead by electro-analysis has been prepared by Eberbach Corporation, Ann Arbor, Michigan, for distribution with its Electro-Analysis Apparatus. The methods and extensive bibliography are a product of research at Battelle Memorial Institute. Because this booklet can be helpful to all laboratories performing such analyses, copies are available upon request at no charge.

Hellige, Inc., 3718 Northern Boulevard, Long Island City, N. Y., has released two new catalogs on apparatus for Precise pH Control and Water Tests. Catalog No. 600-10 illustrates and describes the Hellige Colorimetric Comparator line which ranges from the inexpensive Simplex Testers to the versatile Pocket and Standard Comparators, and the Aqua Tester. All models are equipped with Non-Fading Glass Color Standards, and many new standards are now offered for the latest tests employed in analyses of water, sewage, and industrial waste. The catalog also introduces the Hellige Daylite Comparator Illuminator with which the Pocket and Standard Comparators can be used for determinations in both artificial and day light. A four-color catalog No. 600-20 lists 180 indicator solutions and reagents for pH measurements, water and chemical analyses with the Hellige Comparators.

A calendar intended for those interested in low-temperature research, particularly in the liquid helium temperature range, is available from Arthur D. Little, Inc., 30 Memorial Drive, Cambridge, Mass. A conversion chart of the boiling points of gases is printed on this calendar. Gases from Carbon Tetrachloride to Helium are listed by boiling point in Centigrade, Fahrenheit and Absolute scales.

A new 24-page catalog on "Laboratory Gases and Equipment" is available from the Ohio Chemical & Surgical Equipment Co., 1400 E. Washington Avenue, Madison, Wis. Besides prices, the booklet contains information on safety precautions, basic properties and principal impurities of gases, data on standard valves and cylinders.

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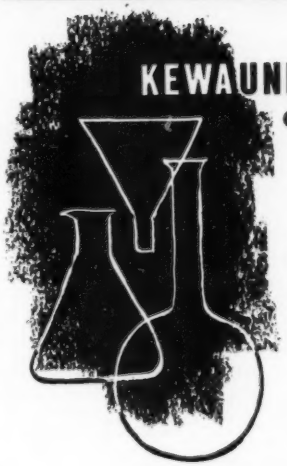

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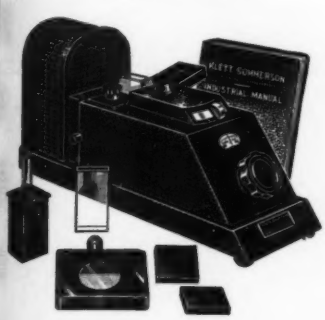

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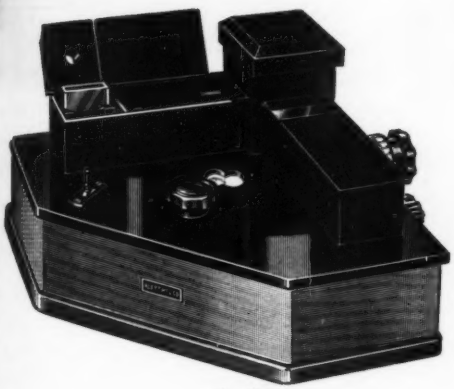

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Remsen's greatest fame was the discovery of the synthesis of saccharin which was the result of the joint work of Fahlberg, a German student working with Remsen and of Remsen himself. The way in which Fahlberg succeeded in concentrating all the fame on himself and in which he commercialized this invention is told in detail and it deserves to be told. It was an unpleasant affair that caused Remsen much trouble even in quarters where he had not expected it, as subsequent difficulties with the editor of the *Journal of the Society of Chemical Industry* proved.

Remsen's work as a university president and a public servant constitute another example of the vicissitudes of an honest scientist in local and national politics and they are well told. Seeing the elaborate bibliography of Remsen's publications appended to this book we have the feeling, even after the pleasant hours spent in reading it, that most has not been made of the evolution of Remsen's practical and theoretical work as seen against the background of American organic chemistry of those days. Should anyone later attempt to write this more elaborate biography he will surely find this book a handy collection of facts to start from.

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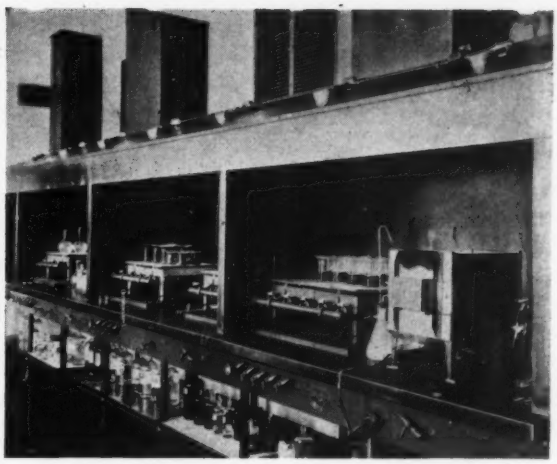

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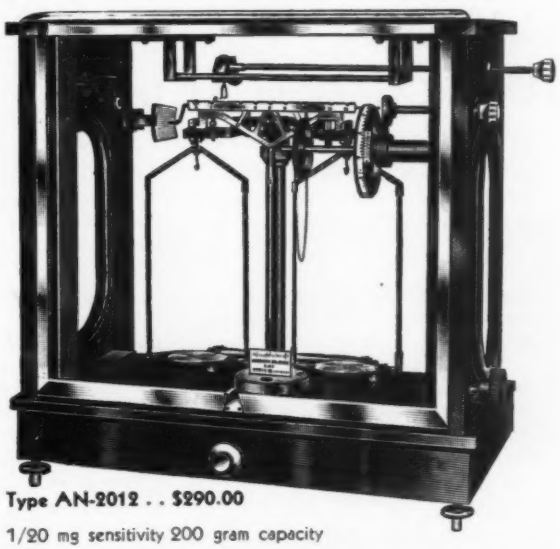

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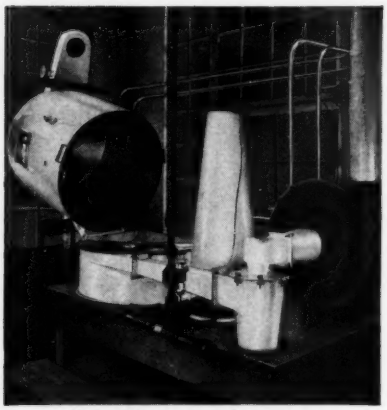

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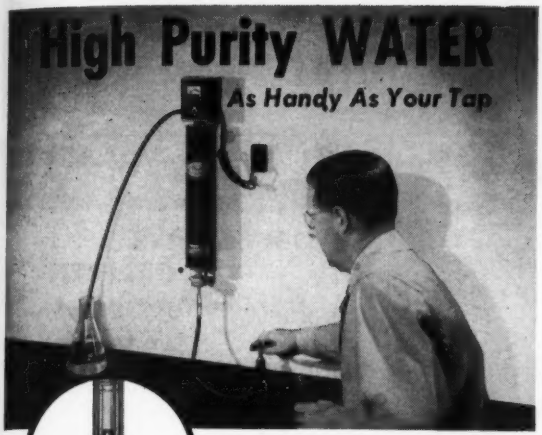

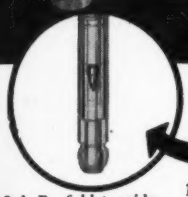

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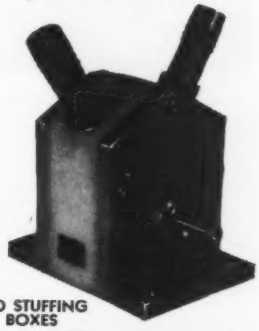

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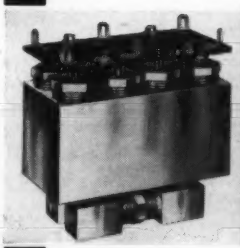

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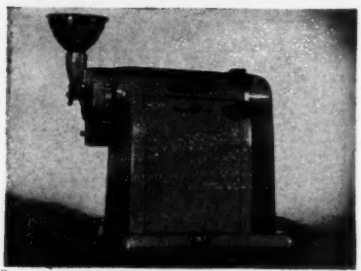

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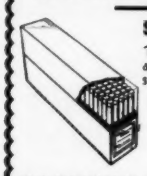

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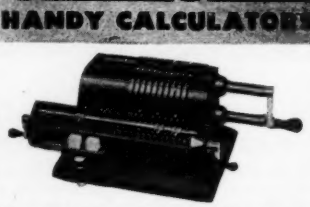

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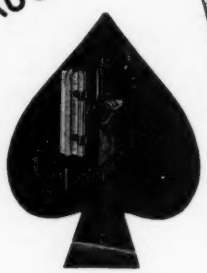

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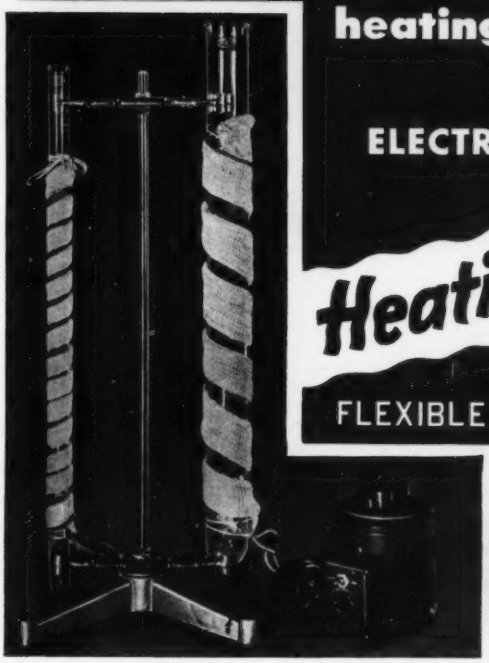

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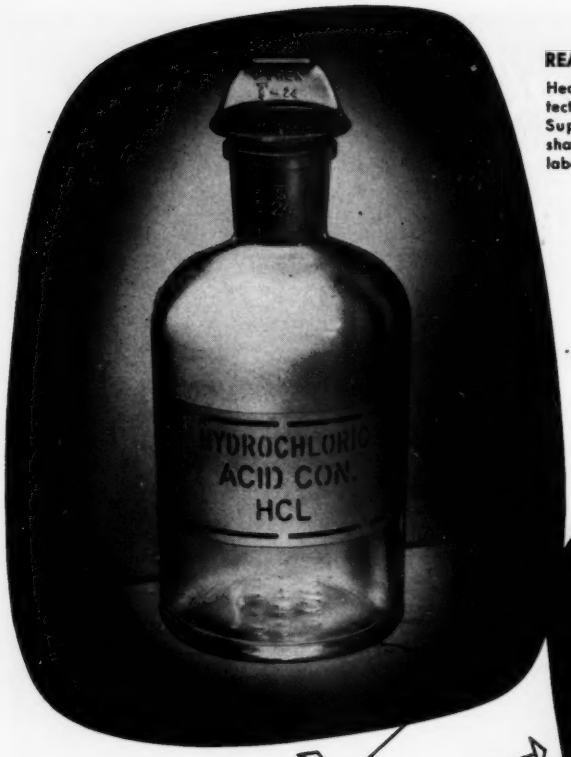

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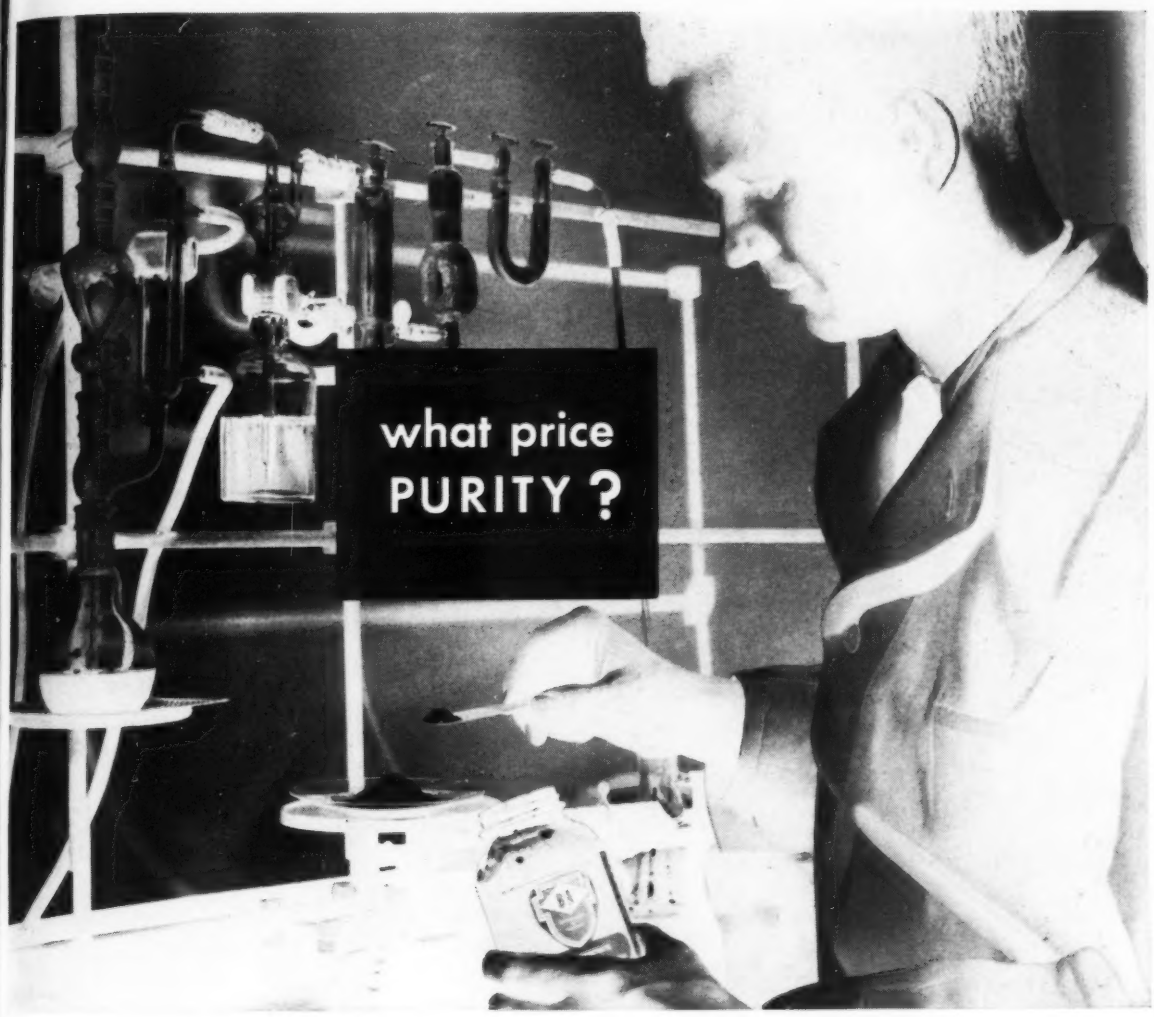

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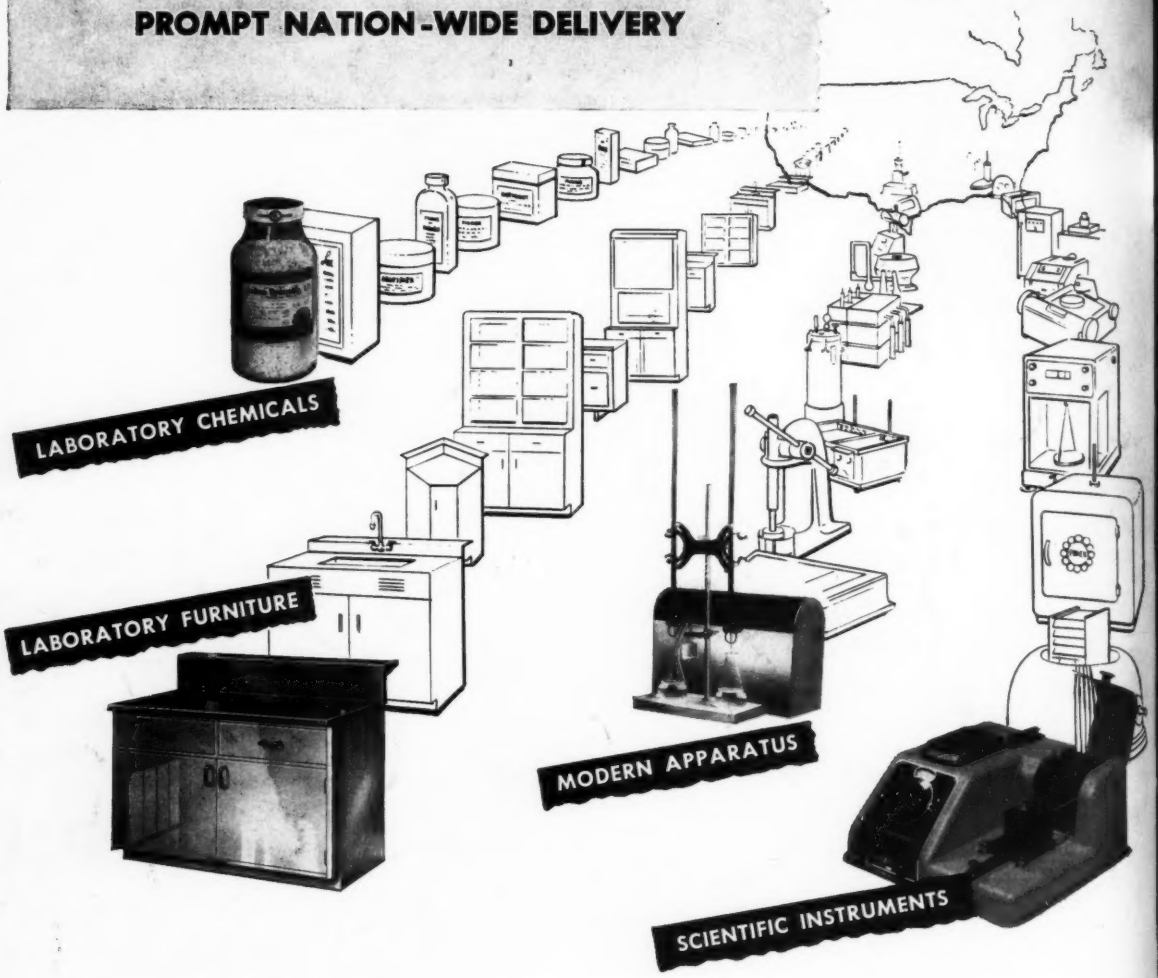

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